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(71) Applicant: **TAKASAGO INTERNATIONAL CORPORATION** [JP/JP]; 37-1, Kamata 5-chome, Ohta-ku, Tokyo, 1448721 (JP).

(72) Inventors: **LOOFT, Jan**; Takasago Europe GmbH, Industriestrasse 40, Zuelpich, 53909 (DE). **KLEINECKE, Klaus**; Takasago Europe GmbH, Industriestrasse 40, Zuelpich, 53909 (DE).

(74) Agents: **KOBAYASHI, Hiroshi** et al.; ABE, IKUBO & KATAYAMA, Fukuoka Bldg. 9th Fl., 8-7, Yaesu 2-chome, Chuo-ku, Tokyo, 1040028 (JP).

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(54) Title: PROCESS FOR PREPARING NATURAL FLAVORS

(57) Abstract: The present invention relates to a process for the preparation of natural flavors from individually quick frozen (IQF) fruits, vegetables and herbs. In particular, authentic, thermally unchanged flavor distillates can be prepared through distillation, on a Spinning Cone Column (SCC), of purees which are freshly prepared from frozen produce, e.g. IQF (individually quick frozen) fruits.



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DESCRIPTION

Title of Invention

Process for preparing natural flavors

Technical Field

The present invention relates to the preparation of natural flavors from individually quick frozen (IQF) fruits, vegetables and herbs.

Background Art

For some years already, a growing trend can be observed: consumers are suspicious of "artificial" products and chemicals. They prefer products which are identified as being "natural", are produced by traditional processes and appear to be "sustainable". This trend also extends to the production of foodstuff by wholefood shops and has resulted in major growth of the organic foods sector.

The trend is also reflected in consumer demands that are addressed at the flavor industry: flavors are preferred, which have an authentic flavor profile, ideally equivalent to the fresh, unprocessed prototype in field crops and cuisine. Artificial flavorings are avoided, and food retailers urge to keep products free of labels which indicate food additives or flavors. This trend runs parallel to regulations originating from the European Commission and national Authorities.

As part of the trend, new categories of flavors, like "from the named fruit" ("FTNF") and "from the named juice" ("FTNJ"), have appeared, and threshold values have been set for additives or auxiliary materials which originate from the processing method.

For example, European Council Directive 88/344/EES specifies a threshold for methanol backlog in food of 10 mg/kg if methanol has been used as solvent for the purpose of extraction.

In contrast, naturally occurring methanol in fruits (originating from pectins within the fruits) is not limited by this directive in any way. However, it is favourable to lower the amount of this naturally occurring methanol as much as possible.

In the same way, Interpretive Guideline No. 2021-01 on Proposition 65 by the California Environmental Protection Agency exempts naturally occurring methanol which is formed by the degradation of pectins in fruits and vegetables. However, methods to lower the methanol content in flavors derived from fruits and vegetables are permitted under proposition 65.

In view of the above, there is a need for flavors which originate from the named fruit for the flavoring of beverages, dairy products and fruit preparations. Furthermore, as was alluded to above, the sensory properties of such flavors should be as close as possible to those of fresh fruit, without displaying degradation or a cooking flavor.

The methods commonly employed to derive flavors as byproducts from the fruit juice industry are based on high volume vacuum concentration processes, which serve to concentrate the juice materials to high brix. However, such processes usually lead to a high degree of thermal damage of the condensed vapors containing the flavor materials, thereby resulting in flavor mixtures which do not have a natural, fresh character, but exhibit thermal damage and a cooked note.

More natural and authentic flavors can be isolated by a special distillation technology called spinning cone column (SCC). This technology, which has been known for some time, combines thin film distillation technologies, extraction of slurries and stripping technologies. If fruit, vegetable or herb purees are used as starting materials on SCC, fresh and authentic flavors can be isolated.

In order to address such issues, Non-patent document 1 makes use of SCC technology:

Several commercially available purees and water phases were distilled by SCC to produce fresh and natural fruit, vegetable and herb flavors. As concerns the character of freshness, these flavors already proved to be superior to commercial flavors prepared by traditional juice processing. However, it was found that concentrates from fruit juices or water phases can contain higher than natural levels of methanol due to the fruit processing causing an increase in methanolysis of pectins. Therefore, it was concluded that commercially available purees and water phases do not represent the ideal starting material for the desired process.

Next, the effect of preparing purees immediately prior to distillation was examined. Purees were produced by means of a cutting machine based on a rotor stator principle, e.g. a model offered by the company "Urschel". Flavors produced by SCC from such purees were found to be even more fresh and authentic in character than flavors made from commercial purees or water phases.

However, the use of fresh produce poses the problem of the production schedule being dependent on such factors as the timing of the harvest (availability of the fruit), the need to keep the produce fresh during transport to the production facility, and ensuring timely processing of the produce.

From a standpoint of facilitating processes upstream to the step of distillation on a SCC column, it was thus decided to investigate the use of frozen produce as starting material, since frozen product can easily be stored.

Initial tests, which used a cutting machine to cut up individual quick frozen (IQF) fruits, were soon found to pose several issues. Firstly, the still frozen solid mass which was produced by the cutting machine was found to block the outlet system of the cutting machine after some time – in the case of frozen strawberries, for example, the outlet system of the cutting machine was blocked after 20 minutes. Furthermore, normal pumps, such as compressed air diaphragm pumps, were not able to pump the still frozen mass, thereby preventing immediate processing thereof on the SCC. And lastly, melting the mass in a stirred vessel for several hours at room temperature, with or without the addition of warm water, in order to allow the mass to be pumped, led to thermal degradation of flavor components and to an increase in methanol content due to enzymatic degradation.

In an attempt to overcome these problems, a setup comprising a sequence of a progressive cavity pump with heated pump head, one or more double pipe (tube) heat exchangers, a rotor/stator cutting machine, a pneumatic pump, a stirred vessel as a buffer and a SCC was devised for carrying out the process.

It was found that using hot water of 90°C as heat transfer medium in the heat exchangers allowed for the continuous production of up to 100 kg/h of thawed fruit purees from IQF fruit without the occurrence of significant thermal damage. However, higher feed rates of around 200 kg/h resulted in insufficient thawing of the puree along with clogging (by freezing) of the pipes and pumps.

Non-patent document 2 describes the use of film technologies in fruit processing in order to recover high quality fruit aromas from purees and juice.

Further, Patent document 1 relates to luxury drink flavors, their manufacture, food containing the flavors, and a method for enhancing flavor of food using the flavors. The flavors are manufactured by (A) subjecting a slurry of luxury drink materials, e.g. coffee beans, tea leaves, etc., to gas-liq. countercurrent distillation to recover aroma components, (B) adsorbing flavor components contained in the distillation residue to synthetic adsorbents, (C) desorbing the adsorbed components using solvents, and, optionally, (D) mixing the recovered extract with the aroma components.

Non-patent document 3 describes the Spinning Cone Column as a multi-stage, agitated plate, gas-liquid contacting device with the ability to handle liquids and slurries of milled vegetables or animal substances. The advantages of the SCC in relation to other more conventional stripping equipment are described as lying in its very low residence times and reduced pressure drops, which make the SCC more energy efficient and allow recovery of flavors from liquids or solid-in-liquid streams with a minimum of damage and with few or no artifacts.

Patent document 2 relates to a process for making a flavoring compositions suitable for use in a food product, e.g. a beverage, a desert, or a noodle soup, etc., wherein the process includes preparing an ion exchange solution containing 0.01-3 wt. % of natural gum, and having a solution viscosity of 5-100 mPa·s, mixing 3-15 wt. % of a milled solid

material, e.g. tea, coffee bean, dried bonito, having a grain size of 60-mesh pass ≥ 50 %, and 0-5 wt. % of ethanol to the ion exchange solution, and applying the mixed slurry to a spinning cone column (SCC) at 60-100°C inside the column at a strip ratio of 1-10 % and operation pressure of 0.2-100 kPa.

Patent document 3 relates to the manufacture of concentrated citrus aroma, as well as to flavoring compositions and foods, e.g. beverages, (frozen) desserts or baked confectionery, comprising the aroma. The aroma is manufactured by continuous vacuum concentration of fruit juice to 100 to 150-fold strength (based on the aroma component) and to an alcohol content of 1-7 wt. %, subsequent removal of essential oil by filtration in the presence of filtration aids, and final separation using a spinning cone column at a column temperature of 40-80°C, a strip ratio of 1-10%, and pressure of 0.2-40 kPa. The method is said to allow water-soluble low-molecular-weight components (e.g., acetaldehyde) to be efficiently concentrated.

Citation List

Non-Patent Literature

- Non-patent document 1: Riley P. C., Sykes S. J.; Industrial Applications of spinning cone column technology: a review. *Distillation and Absorption*, Baden Baden, October 2002.
- Non-patent document 2: Skaliotis, L.; *Fruit Processing* (2011), 21(5), 182-186.
- Non-patent document 3: Miyazaki, K. M.; *Aroma Research* (2007), 8(2), 120-124.

Patent Literature

- Patent document 1: JP 2010 259364 A
- Patent document 2: JP 2001 172667 A
- Patent document 3: JP 2001 152180 A

Summary of the Invention

Technical Problem

In view of the above facts, the present inventors have made it their goal to devise a process for producing natural flavors which have flavor profiles that are as close as possible to those of the natural product raw materials and do not exhibit cooked or processed notes.

Furthermore, it was desired to avoid the presence of high levels of methanol in the flavors, as it is impossible to later on separate methanol from the flavor fractions, e.g. by

distillation or through reverse osmosis, without causing the loss of important flavor components.

Lastly, from the standpoint of efficiency and economics, it was desired to devise a process which would be capable of continuously processing large quantities of raw materials.

Solution to Problem

As part of the efforts to achieve the goals described above, the inventors have discovered that authentic, thermally unchanged flavor distillates can be prepared through distillation, on a Spinning Cone Column (SCC), of purees which are freshly prepared from frozen produce, e.g. IQF (individually quick frozen) fruits.

In particular, it was discovered that flavors exhibiting a very low methanol content can be produced by a process which makes use of a scraped surface heat exchanger to defrost the frozen produce. Such a process can be continuously carried out at high feed rates without causing damage, such as fouling or blocking, to the heat exchanger and proves to be highly energy efficient.

Thus, the present invention comprises the following:

Item 1: A process for the production of a flavor, comprising the steps of

- (i) thawing frozen fruit, vegetable or herb by means of a scraped surface heat exchanger to obtain a slurry,
- (ii) optionally processing the slurry by means of a cutting device to obtain a puree, and
- (iii) distilling the slurry or puree by means of a spinning cone column.

Item 2: The process according to item 1 wherein the processing of the thawed fruit, vegetable or herb by means of a rotor/stator type cutting device (step (ii)) is carried out.

Item 3: The process according to item 1 or item 2 wherein the frozen fruit, vegetable or herb is individual quick frozen (IQF).

Item 4: The process according to item 3 wherein the individual quick frozen fruit, vegetable or herb is individual quick frozen fruit.

Item 5: The process according to item 4 wherein the individual quick frozen fruit is individual quick frozen strawberry, blueberry or mango.

Item 6: The process according to any one of items 1 to 5, which further makes use of a progressive cavity pump for feeding the frozen fruit, vegetable or herb to the scraped surface heat exchanger.

- Item 7: The process according to item 6, wherein the progressive cavity pump is equipped with an integrated heat exchanger.
- 5 Item 8: The process according to any one of items 1 to 7, wherein the process is carried out as a continuous or a batch process.
- Item 9: The process according to item 8, which is carried out as a continuous process.
- 10 Item 10: The process according to any one of items 1 to 9, wherein the mean temperature of the fruit, vegetable or herb mass does not exceed 30°C prior to distillation by the spinning cone column.
- 15 Item 11: The process according to any one of items 1 to 10, wherein the temperature of the heating medium in the scraped surface heat exchanger lies within the range of 30 °C to 70 °C.
- 20 Item 12: Use of a flavor prepared according to the process of any one of items 1 to 11 for flavoring beverages, dairy products or fruit preparations.
- Item 13: An apparatus for carrying out the process of any one of items 1 to 11, comprising a scraped surface heat exchanger, a cutting device and a spinning cone column.
- 25 Item 14: A process for the thawing of frozen fruit, vegetable, or herb, which is characterized by the use of a scraped surface heat exchanger.
- 30 Item 15: The process according to item 14, which yields a fruit, vegetable or herb slurry.
- Item 16: A process for the production of a puree, which is characterized in that a fruit, vegetable or herb slurry which is produced according to item 15 is further processed by means of a cutting device.
- 35 Item 17: The process according to item 16, wherein the puree has a mean temperature of 0 to 30 °C.

40 **Brief Description of Drawings**

Figure 1 shows the MeOH Content in Strawberry SCC distillates.

Figure 2 shows the MeOH Content in Blueberry SCC distillates.

45

Figure 3 shows the MeOH Content in Mango SCC distillates.

Description of Embodiments

5 With the above-stated goals in mind, the present inventors investigated the use of a scraped surface heat exchanger for processing the frozen raw materials, such as IQF fruit.

Scraped surface heat exchangers originally were designed for the processing of whole
pieces of fruit, having diameters up to 2.5 cm. Thus, scraped surface heat exchangers
10 have, in the past, been employed for the pasteurization of non-frozen fruits including fast cooling of the resulting pasteurized mass to lower temperatures, which generally lie above freezing.

For example, TERLET, the manufacturer of the TERLOTHERM® brand of scraped surface
15 heat exchanger, mentions in their documentation the possibility of carrying out deep-cooling and crystallization operations. However, the defrosting of IQF fruit or individual pieces of fruit by use of scraped surface heat exchangers has not previously been described. Also, previous publications do not provide any information on how the continuous thermal thawing of fruits without thermal damage might be carried out.

20 While previously known uses of scraped surface heat exchangers include the freezing of highly viscous liquid materials, the thawing of materials or, for that matter, of heterogeneous materials bearing larger particles having a mean diameter of several cm is not described. In view thereof, it was unclear whether bigger pieces of frozen fruit could
25 be handled inside of a scraped surface heat exchanger without problems. In particular, the following complications were anticipated:

- a) blocking of the stirred scraper system
- b) damage to the scrapers
- 30 c) damage to the inner and outer walls of the heat exchanger by frozen particles
- d) local overheating of fruit material on blocked surfaces of the heat exchanger and consequent thermal degradation of flavor.

In order to investigate the possibility of using a scraped surface heat exchanger to thaw
35 the IQF produce, a setup comprising a sequence of a progressive cavity pump with heated pump head, a Terlotherm® scraped surface heat exchanger, a rotor/stator cutting machine, a pneumatic pump, a stirred vessel as a buffer and a SCC was devised.

Using this setup, it was possible to process frozen fruit having an initial temperature
40 ranging from -5 °C to -20 °C at feed rates of up to 650 kg/h. Furthermore, it was found that the temperature and feed rate of the heat transfer medium heating the scraped surface heat exchanger could be adjusted so as to allow the mean temperature of the material exiting the scraped surface heat exchanger to be above its freezing point (i.e., it is thawed) and the mean temperature of the fruit puree exiting the cutting machine to lie
45 between 30 °C and 0 °C, and preferably between 20 °C and 10 °C. This represents a significant advantage, since any blockage due to the freezing of pipes, pumps, or the

cutting device, which was experienced with the process employing conventional heat exchangers at lower feed rates of 200 kg/h, is eliminated, thereby making it possible to attain much higher throughput rates, which can be continuously maintained.

Furthermore, being able to provide a continuous feed to the SCC allows for steady operating conditions, thereby further enhancing the process efficiency.

In the process of the present invention, the temperature of the heating medium in the scraped surface heat exchanger generally lies within the range of 30 to 70 °C, preferably in the range of 40 to 60 °C. In particular, the process employing the scraped surface heat exchanger has the advantage of allowing heating water of significantly lower temperature (e.g. 55°C) to be used. In contrast thereto, a heating water temperature of 90°C was required for the process employing conventional heat exchangers, thereby increasing the risk of local thermal damage and flavor degradation as well as the chance of an increase in methanol content due to degradation of pectins. The much gentler thawing of the frozen produce which is possible with the process of the present invention thus allows flavor distillates to be produced, which are more authentic than those which can be obtained by previous processes.

For purposes of balancing the goals of process efficiency and good heat exchange on the one hand with the requirement of avoiding thermal damage and flavor degradation on the other hand, the scraped surface heat exchanger in the process of the present invention is preferably operated at a rotational speed lying in the range of 100 to 150 rpm, with a rotational speed of about 120 rpm being most preferred.

A comparison of the flavor of the distillate obtained by the new process to equivalent flavors from commercial distillates and to a distillate obtained from frozen produce by means of processes lacking a heat exchanger or employing a conventional double pipe heat exchanger (including a step of thawing in a stirred vessel) showed the flavor to be more fresh and to exhibit less signs of thermal damage.

Methanol content of the processed material

Some commercial flavors suffer from very high methanol levels. In view thereof, it would be desirable for the new processes to allow the production of comparable flavors having lower methanol levels.

Analysis of the methanol content of the flavor distillates which were produced by the different methods showed that distillates obtained from commercial water phases and purees can contain high levels of methanol. This is likely due to the fruit processing causing an increase in methanolysis of pectins. Furthermore, a marked increase in the methanol content of the distillates was also observed when letting a freshly prepared puree stand for a prolonged period of time prior to further processing it.

In contrast thereto, the newly developed process overcomes all of these issues by enabling the puree to be produced in the same amount as can be fed to a SCC. This allows for continuous processing of the puree to be carried out immediately after its production, thereby limiting the amount of time that is available for methanol formation.

- 5 Furthermore, the new process also precludes methanol by avoiding thermal overheating during thawing. Thus, the new process allows the preparation of fresh and authentic flavors which have a very low methanol content.

10 Examples

Methods and Equipment

15 General experimental setup and procedure for the processing of IQF-fruit using a double pipe heat exchanger (Process 1):

- Progressive cavity pump, heated pump head (Pump by company "Knoll", Knoll MX 30R 60/10)
- Two double pipe heat exchangers, 2 m length, 3,8 cm inner diameter
- 20 - Rotor/stator cutting machine
- Pneumatic pump
- Stirred vessel as a buffer, volume 600 l
- SCC M1000

- 25 A progressing cavity pump, produced by Knoll, type MX 30R-60/10 with heated pump head was equipped succinct with a feed hopper 80 cm high. Frozen but free rolling fruit was filled into the hopper to a height of 60 cm. The feed hopper was replenished during fruit processing.

- 30 The temperature of the frozen fruit, the rotation speed of the screw (scrapers), and the inlet- and outlet temperatures and flow rate of the heating media were as indicated in the specific examples.

- 35 The outlet of the progressive cavity pump was connected via a hose to two double pipe heat exchangers in one row. The heat exchangers were made in-house from stainless steel. The diameter of the inner pipe was 3.8 cm and the length of each heat exchange surface was 2.0 m. The heat exchangers were operated with hot water in counter-current flow as heating media. Afterwards this medium was used to heat the pump head of the progressing cavity pump.

- 40 Temperatures of the heating media were measured at the inlet and outlet of the heat exchanger. The measured temperatures are indicated in the specific examples along with the temperature of the frozen mass and the overall flow rate of hot water.

The outlet of the heat exchanger was connected to the inlet of a cutting machine by means of a flexible hose. A Cutting machine manufactured by "Urschel", type Comitrol 1500, equipped with cutting head 3M-025040U 66866 was used.

5 The discharge from the cutting machine was collected in a 40 l bucket below the outlet of the machine. In some cases the outlet of the cutting machine was blocked by frozen puree after some time (see indication in specific examples). Continuous pumping of the puree by an air operated diaphragm pump was hampered by freezing of the pumps. Consequently, the bucket was emptied by hand into a stirred 600 l vessel.

10 The mass was maintained in the vessel under stirring to allow thawing and dilution, if required. Subsequently, it was processed on the SCC. The distillation conditions (Top temperature on distillation column, Offset-temperature, total strip rate, total flow of puree) are indicated in the specific examples.

15 The thawing time of the freshly prepared purees was used to investigate the influence of storage time on the release of methanol. The corresponding data is listed with the specific examples.

20 **General experimental setup and procedure for the processing of IQF-fruit by use of scraped Surface heat exchanger (Process 2):**

- Progressing cavity pump, heated pump head (Pump by company "Knoll"):
25 type MX 30R-60/10)
- Terlotherm® Scraped Surface Heat exchanger by company Terlet, Terlotherm® T0-4.
- Cutting machine, by company "Urschel", Comitrol 1500, Rotor/Stator, type cutting device: 3M-025040U 66866 and self-constructed, closed outlet funnel
- Pneumatic pump, membrane type, type P4
- 30 - Stirred vessel as a buffer, volume 600 l
- SCC, company "Flavourtech", type SCC 1000

35 A progressing cavity pump, produced by Knoll, type MX 30R-60/10 with heated pump head was equipped succinct with a feed hopper 80 cm high. Frozen but free rolling fruit was filled into the hopper to a height of 60 cm. The feed hopper was replenished during fruit processing.

40 The temperature of the frozen fruit, the rotation speed of the screw (scrapers), and the inlet- and outlet temperatures and flow rate of the heating media were as indicated in the specific examples.

45 The outlet of the progressive cavity pump was connected via stainless steel tubing to the bottom inlet of a Scraped Surface Heat exchanger (manufactured by Terlet, type Terlotherm® T0-4). The surface temperature of this 30 cm tubing was measured by a Pt-100 sensor. Measurements inside of the tubing were avoided in view of the high shear

forces exerted by the frozen mass and to avoid blocking of the tubing. The measured temperatures are indicated in the specific examples.

The Terlotherm® heat exchanger was operated with hot water in counter current flow as heating media. Afterwards this media was used to heat the pump head of progressing cavity pump. Temperatures of heating media were measured at the inlet and outlet of the Terlotherm.

The outlet of the scraped surface heat exchanger was connected to the inlet of a cutting machine by means of a flexible hose. A Cutting machine manufactured by "Urschel", type Comitrol 1500, equipped with cutting head 3M-025040U 66866 was used.

The discharge from the cutting machine was collected by means of a self-constructed, closed outlet funnel and pumped down to a stirred 600 l vessel by means of an air operated diaphragm pump.

The surface temperature of the outlet funnel is indicated in the specific examples.

The flow of hot water as heating medium was started and the progressing cavity pump and scraped surface heat exchanger were filled with water before the IQF fruits were processed.

At the beginning of the trial 90 kg of IQF fruit were processed and the resulting puree discarded in order to establish a temperature equilibrium in the setup.

Distillation on a spinning cone column (SCC) (manufactured by Flavourtech, M1000, model SCC 1000) was started once a sufficient amount of puree was accumulated in the stirred buffer vessel. The distillation conditions (Top temperature on Distillation column, Offset-temperature, total strip rate, total flow of puree) are indicated in the specific examples.

Samples of distillates were collected, their methanol content measured by GC and their flavor evaluated sensorically through examination by three sensorically trained persons (Flavorists).

General experimental procedure for the processing of fresh fruit and the distillation of freshly prepared puree on the SCC (Process 3):

A progressing cavity pump, produced by Knoll, type MX 30R-60/10 was equipped succinct with a feed hopper 80 cm high. Fresh, free rolling fruit was filled into the hopper to a height of 60 cm. The feed hopper was replenished during fruit processing.

The outlet of the progressing cavity pump was connected to the inlet of the cutting machine via a flexible hose. A Cutting machine manufactured by "Urschel", type Comitrol 1500, equipped with cutting head 3M-025040U 66866 was used.

The discharge was collected by a self-constructed, closed outlet funnel and pumped down to a stirred 600 l vessel by use of an air operated diaphragm pump.

- 5 Distillation on a spinning cone column (SCC) (manufactured by Flavourtech, M1000, model SCC 1000) was started immediately once a sufficient amount of puree was accumulated in the stirred buffer vessel. The distillation conditions (Top temperature on Distillation column, Offset-temperature, total strip rate, total flow of puree) are indicated in the specific examples.

10

Samples of distillates were collected, their methanol content measured by GC and their flavor evaluated sensorically.

15 **Determination of methanol content:**

- The methanol content of the SCC distillates was determined by GC. When determining the methanol content of natural extracts by GC, a problem of overlap with other compounds in the methanol retention range, such as ethyl acetate, acetal, or mixed Acetals, can arise. In order to avoid this problem, a GC x GC/MSD System was used: Precolumn BCWax 50m x 0.25mm x 0.15µm, cut to Restek RT-β-DEXse 30m x 0.25mm x 0.25µm.

25 **Example 1**

The setup of process 2 was employed to process the following IQF materials:

- 30
- IQF Whole Strawberries "Camarossa", Morocco, uncalibrated
 - IQF Strawberries "American No. 13", China, uncalibrated
 - IQF Strawberries "Senga Sengana", Poland, uncalibrated
 - IQF Blueberries (not cultured), Lithuania
 - IQF Mango Diced "Kent", 20 x 20 mm, Peru

- 35 Parameters for processing of IQF-fruit by use of scraped surface heat exchanger, and SCC distillation conditions and evaluation for scraped surface heat exchange processed IQF-fruit are shown in Tables 1 and 2, respectively.

Table 1 (parameters for processing of IQF-fruit by use of scraped surface heat exchanger)

IQF material	Temp. fruit °C	Flow fruit kg / h	Flow heating media l/h	Knoll heating out °C	Knoll heating in °C	Outlet funnel °C	Terlet heating in °C	Terlet heating out °C
Strawberry, Camarossa	-12,2	500	5000	40	43	24	55	50
Strawberry, American No. 13	-12	440	4700	43	45	23	58,5	52,6
Strawberry, Senga Sengana	-12	580	4800	42	44	18	57	51
Wild Blueberry from Lithuania	-12	640	5400	38	39	23	39	38
Mango chops, Peru	-12,6	580	2800	43	46	24	63	53,4

Table 2 (SCC distillation conditions and evaluation for scraped surface heat exchange processed IQF-fruit)

IQF material	Top Temp °C	Offset °C	Ext. Strip rate %	Flow l/h	Dilution	MeOH % (w/w)	Description
Strawberry, Camarossa	80	-10	3	400	Pure	0,006 - 0,008	Fruity, fresh, ripe
Strawberry, American No. 13	80	-10	3	500	Pure	0,011 - 0,023	Fresh, seedy, fruity
Strawberry, Senga Sengana	60	-10	3	450	Pure	0,021	Impact, green, acidic
Wild Blueberry, Lithuania	100	-10	10	400	water 1:1	0,028	Fruity, dry, dusty,
Mango chops, Peru	60	-10	3	400	water 2:1	0,017	Green & cucumber, pulpy & terpenic

Comparative Example 1

- 5 The setup of process 1 was employed to process strawberries and blueberries.

Processing of IQF Strawberry Senga Sengana from Poland.

1.) Preparation of puree from Strawberries, -5.5°C

- 5 640 kg of IQF Strawberries "Senga Sengana" were stored in a cooling chamber at 4 °C overnight to reach a mean temperature of -5.5°C prior to the experiment.

The conditions indicated in entry 1 of Table 3 were used.

- 10 After 30 min the membrane pump was blocked and a second pump was added.
After 1 hour the experiment had to be interrupted because the inner side walls and the outlet of the cutting device were blocked by freezing and material flooded the cutting device.
35 minutes after restarting the experiment, both membrane pumps were blocked again.
15 Consequently, it was not possible to establish a continuous production rate of >300 kg/h.

2.) Preparation of puree from Strawberries, -14°C

- 20 410 kg of Strawberries at -14°C were used directly in the mentioned setup. To compensate for the lower temperature of the strawberries the flow rate of the heat exchanger medium was increased to 800 l/h, see the conditions indicated in entry 2 of Table 3.
25 After 20 minutes the flow rate decreased significantly due to blocking of the transition piece between the progressive cavity pump and the heat exchanger. 10 minutes later the experiment was interrupted and the transition piece cleaned.
30 20 minutes after restarting the experiment, the membrane pump stopped because of freezing within the pump. A second pump was used, but this membrane pump also stopped operating after 20 minutes.

- Consequently, it was not possible to establish a continuous production rate of >280 kg/h.
35

3.) Preparation of puree from wild blueberries from Poland, -4,5°C

- 40 510 kg of IQF wild Blueberries from Poland were stored in a cooling chamber at 4°C overnight to reach a mean temperature of -4,5°C prior to experiment.

The conditions indicated in entry 3 of Table 3 were used.

- 45 This setup worked well for flow rates corresponding to 15% of the power of the progressive cavity pump (200 kg berries /h). However, upon increasing the flow rate of

the progressive cavity pump from 15% to 20% of the power rate, the cutting device was frozen.

Consequently, it was not possible to establish a continuous production rate of >200 kg/h.

4.) Processing of puree on SCC

Half of each of the purees produced according to 1.) and 2.) above was distilled directly on SCC under the conditions indicated in entries 4 and 5 of Table 4, the remaining puree was stored overnight in a cooling chamber at 4°C. During this time, the mean temperature of the puree rose to -0,8°C. This material was distilled on SCC under the conditions indicated in entries 6 and 7 of Table 4.

The puree produced according to 3.) was above 0°C, but thixotropic. It was not possible to continuously process this puree on the SCC without dilution, because of fouling at the SCC feed stock heater. Consequently, half of the material was diluted immediately with water and distilled on SCC under the conditions indicated in entries 8 and 9 of Table 4. The other half of the material was stored in a cooling chamber at 4°C overnight and then distilled on SCC under the conditions indicated in entries 10 and 11 of Table 4.

5.) Methanol content of strawberry distillates

The methanol content of the distillates was determined by the general procedure described above. Although the puree was kept at temperatures below 0°C the entire time before processing on the SCC, the methanol content was found to have increased 3-fold when delaying the processing by 24 hours and 5-fold when delaying the processing by 34 hours.

6.) Methanol content of blueberry distillates

The methanol content of the distillates was determined by the general procedure described above. Although the stored puree was kept at temperatures below 4°C overnight, the methanol content within the distillates doubled in comparison to distillates produced from freshly prepared puree.

Table 3 (parameters for processing of IQF-fruit by use of double pipe heat exchanger)

Entry	Fruit(IQF)	Temp. fruit °C	Flow fruit kg / h	Flow heating media l/h	Knoll heating in °C	Heat exchanger outlet °C	Heat exchanger heating out °C	Temp. Puree
1	Senga Sengana	-5,5	330	500	90	-2	50	-1,8
2	Senga Sengana	-14	280	800	94	-2,5	50	-1,7
3	Wild blueberry from Poland	-4,5	200	500	90	2	50	3,5

Table 4 (SCC distillation conditions and evaluation for double pipe heat exchange processed IQF-fruit)

Entry	Fruit(Puree from IQF processed according to Table 3)	Storage time Puree	Top Temp °C	Offset °C	Ext. Strip rate %	Flow l/h	Dilution	MeOH % (w/w)	Description
4	Senga Sengana	Freshly	60	-3	3	500	Pure	0,022	Impact, green, acidic
5	Senga Sengana	Freshly	100	-3	3	500	Pure	0,030	Less green, slightly boiled
6	Senga Sengana	24 h	100	-3	3	500	Pure	0,084	Less green, Slightly boiled
7	Senga Sengana	34 h	100	-3	3	500	Pure	0.160	Less green, slightly boiled, jammy
8	Wild blueberry from Poland	Freshly	60	-3	5	300	1:1	0,030	Herbal, stalky, fruity, green
9	Wild blueberry from Poland	Freshly	100	-10	3	300	1:1	0,043	Herbal, fruity, slightly jammy
10	Wild blueberry from Poland	24 h	60	-3	3	500	1:1	0,080	Herbal, stalky, fruity
11	Wild blueberry from Poland	24 h	100	-10	5	500	1:1	0,068	Fruity, jammy

Comparative Example 2

Preparation of distillates from commercial water phases and purees.

- 5 The raw material was placed directly into a 600 l stirred tank and immediately processed on a spinning cone column (manufactured by Flavourtech, model SCC 1000) according to the conditions (Top temperature on Distillation column, Offset-temperature, total strip rate, total flow of puree) indicated in the table below.

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Table 5 (SCC distillation conditions and evaluation for commercial water phases and purees)

Entry	Raw material	TopTemp °C	Offset °C	Ext. Strip rate %	Flow l/h	Dilution	MeOH % (w/w)	Description
12	Strawberry recovery flavor FTNF	80	-3	4	500	Pure	5,1	Fruity, boiled, jammy
13	Blueberry extract FTNF	60	-10	5	500	Pure	10	Typical
14	Mango recovery flavor FTNF	80	-3	4	500	pure	0,15	Woody, terpenic
15	Strawberry puree, aseptic, 4°C	80	-3	4	500	Pure	0,06	Fresh, fruity, but weak
16	Blueberry puree, aseptic, 4°C	80	-3	3	500	Pure	0,18	Herbal, stinky, fruity, but weak
17	Mango puree Alphonso, 4°C, 17 brix	80	-3	3	500	Pure	0,25	Juicy, terpenic, pulpy, less impact

Reference Example 1

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Preparation of distillates from fresh fruit.

Processing and evaluation was carried out according to the general experimental procedure provided above (process 3).

Table 6 (SCC distillation conditions and evaluation for purees from fresh fruit)

Entry	Raw material	Top Temp °C	Offset °C	Ext. Strip rate %	Flow l/h	Dilution	Description
18	Strawberry Eljana	100	-3	3	500	Pure	light floral, seedy, jammy
19	Strawberry Elsanta	60	-3	3	500	pure	Sweet, fruity, ripe, slight green, seedy
20	Strawberry Elsanta	100	-3	3	500	Pure	Sweet, fruity, ripe seed, slight jammy
21	Strawberry Darselect	60	-3	3	500	pure	Sweet, estery, very ripe, strong berry, green
22	Blueberry, fresh, wild	60	-3	3	500	pure	Green, light fruity, stalky

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Results

10 As is seen from the sensorical evaluation data of the distillates obtained in Example 1 and Comparative Examples 1 and 2 and Reference Example 1, distillates obtained from frozen fruit exhibit a sensory profile which is very similar to that of distillates which are obtained from fresh fruit, i.e. very authentic and green flavors. In contrast thereto, distillates
15 obtained from commercial purees are less fresh in character and distillates obtained from water phases typically exhibit boiled notes.

Furthermore, the effects of the processing conditions can be summarized as follows: Low top temperatures on the SCC result in fresh flavors if the raw material is fresh; high top temperatures on the SCC add boiled and jammy notes, and storage of the freshly
20 prepared puree results in a decline of the green and fresh character.

As concerns the methanol content of the distillates, it can be seen that distillates obtained from commercial water phases and purees contain high levels of methanol. Furthermore, prolonged storage, even at low temperatures leads to a significant increase in methanol content. In contrast thereto, the distillates which are prepared by the newly developed process exhibit a very low methanol content. (See Figures 1 to 3 for a graphical comparison of the methanol content of the flavors obtained in Example 1 and Comparative Examples 1 and 2.)

Thus, it can be seen that the newly developed process allows the preparation of fresh and authentic flavors which exhibit a very low methanol content.

CLAIMS

- Claim 1: A process for the production of a flavor, comprising the steps of
- 5 (i) thawing frozen fruit, vegetable or herb by means of a scraped surface heat exchanger to obtain a slurry,
- (ii) optionally processing the slurry by means of a cutting device to obtain a puree, and
- 10 (iii) distilling the slurry or puree by means of a spinning cone column.
- Claim 2: The process according to claim 1 wherein the processing of the thawed fruit, vegetable or herb by means of a rotor/stator type cutting device (step (ii)) is carried out.
- 15 Claim 3: The process according to claim 1 or claim 2 wherein the frozen fruit, vegetable or herb is individual quick frozen (IQF).
- Claim 4: The process according to claim 3 wherein the individual quick frozen fruit, vegetable or herb is individual quick frozen fruit.
- 20 Claim 5: The process according to claim 4 wherein the individual quick frozen fruit is individual quick frozen strawberry, blueberry or mango.
- Claim 6: The process according to any one of claims 1 to 5, which further makes use of a progressive cavity pump for feeding the frozen fruit, vegetable or herb to the scraped surface heat exchanger.
- 25 Claim 7: The process according to claim 6, wherein the progressive cavity pump is equipped with an integrated heat exchanger.
- 30 Claim 8: The process according to any one of claims 1 to 7, wherein the process is carried out as a continuous or a batch process.
- Claim 9: The process according to claim 8, which is carried out as a continuous process.
- 35 Claim 10: The process according to any one of claims 1 to 9, wherein the mean temperature of the fruit, vegetable or herb mass does not exceed 30°C prior to distillation by the spinning cone column.
- 40 Claim 11: The process according to any one of claims 1 to 10, wherein the temperature of the heating medium in the scraped surface heat exchanger lies within the range of 30 to 70 °C.
- 45 Claim 12: Use of a flavor prepared according to the process of any one of claims 1 to 11 for flavoring beverages, dairy products or fruit preparations.

Claim 13: An apparatus for carrying out the process of any one of claims 1 to 11, comprising a scraped surface heat exchanger, a cutting device and a spinning cone column.

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Claim 14: A process for the thawing of frozen fruit, vegetable, or herb, which is characterized by the use of a scraped surface heat exchanger.

Claim 15: The process according to claim 14, which yields a fruit, vegetable or herb slurry.

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Claim 16: A process for the production of a puree, which is characterized in that a fruit, vegetable or herb slurry which is produced according to claim 15 is further processed by means of a cutting device.

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Claim 17: The process according to claim 16, wherein the puree has a mean temperature of 0 to 30 °C.

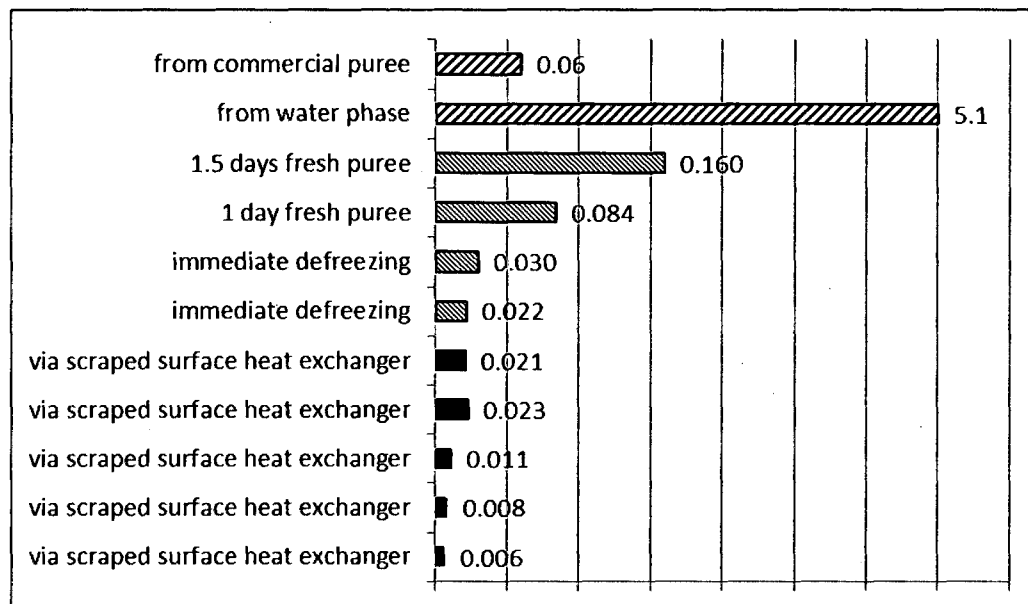
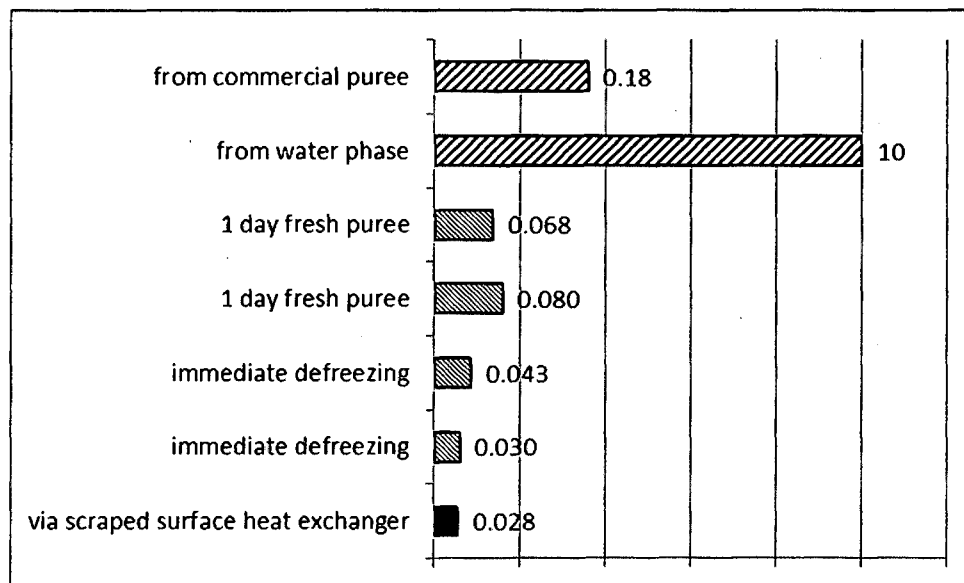
Figure 1: MeOH Content in Strawberry SCC distillates (w/w%)**Figure 2: MeOH Content in Blueberry SCC distillates (w/w%)**

Figure 3: MeOH Content in Mango SCC distillates (w/w%)