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(73) Patenthaver: **Euroserum, Route de Luxeuil-Les Bains, 70170 Port Sur Saone, Frankrig**

(72) Opfinder: **Perceaux, Romain, Résidence Les Bains 1, 1 rue Franche Comté, 70000 Vesoul, Frankrig**
Naegele, Delphine, 2 rue de la fontaine Gaville, 70000 Pusy ET Epenoux, Frankrig
Vareille, Philippe, 15 rue Robert Clave, 70000 Noidans LES Vesoul, Frankrig

(74) Fuldmægtig i Danmark: **Plougmann Vingtoft A/S, Rued Langgaards Vej 8, 2300 København S, Danmark**

(54) Benævnelse: **Salt fra mælk med højt indhold af kalium og fremgangsmåde til opnåelse af samme**

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JP-A- S63 141 561
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The invention concerns primarily a dairy-origin potassium-rich salt.

The invention also concerns the method for obtaining such a salt.

In terms of health, excessive sodium consumption is targeted as being one of the main risk factors of certain diseases, and essentially cardiovascular diseases.

Diet programs recommend reducing the daily intake of salt to cut down the detrimental effects of sodium. These programs are based on studies demonstrating that reducing by half the daily salt intake, to achieve a recommended level of approximately 5 g, will considerably decrease the onset of some cardiovascular diseases.

The commonly used salt based on sodium is found in most foods. Reducing the consumer daily sodium intake is therefore difficult to check. It is estimated that 20% of salt intake can be checked by consumers, essentially by reducing table salt consumption. However, if the reduction, or even the elimination, of table salt reduces the ingested sodium content, this elimination is to the detriment of taste.

Potassium salts with a reduced sodium content and a potassium-rich content have appeared recently. These salts can serve as a substitute for traditional sodium salt while preserving a salty taste thanks to the presence of potassium.

However, these potassium salts more often than not maintain a high sodium content or appear in a powdery form which does not encourage consumers to use it as a table salt substitute.

This is particularly the case of Lactosalt® which comes in two different forms. Lactosalt Optitaste® has a sodium content of 8% and appears as a non-crystallized powder, whereas Lactosalt 27M has a high 27% sodium content.

Lactoserum is a cheese-making co-product whose dry extract mineral salts contain approximately 40% potassium, 12% sodium and a 28% chloride. Accordingly, lactoserum is naturally rich in potassium and the presence of chloride makes it suitable for use in producing a potassium-rich salt.

Using lactoserum to manufacture a potassium-rich salt is known in particular from publication EP 1 061 811 which proposes a method for producing lactoserum salt powder consisting essentially in lactoserum nanofiltration or of lactoserum ultrafiltration permeate, followed by a nanofiltration permeate concentration step and a drying step.

With this method, a salt powder can be obtained differing substantially in appearance from traditional table salt. For the reasons mentioned previously, the consumer or manufacturer therefore is less likely to replace table salt by this salt powder.

A crystallized salt of daily origin is also known from publication EP 0 536 612.

Another substitute for dairy origin salt is marketed under the brand name of Lactosalt® Optitaste® which has a sodium content of 8% but also appears in powder form.

In this context, this invention aims at the generation of a dairy origin salt, rich in potassium and which mitigates the aforementioned drawbacks. The invention also aims at a method of producing such a salt.

For this purpose, the dairy origin salt of the invention is crystallized, with a composition having a ratio between the potassium content and the sodium content included between 3 and 6, and more than 50% of the crystals of this salt having an average diameter of more than 100 micrometers.

The dairy origin salt of the invention may also embody the following optional characteristics considered on their own, or in any possible technical combination:

- the ratio between the potassium content and the sodium content is included between 4 and 6.

- the composition includes, with the contents expressed as mass percentages:

  $35\% \leq K \leq 45\%$

  $7\% \leq Na \leq 11\%$

  $45\% \leq Cl \leq 55\%$

  $Ca \leq 0.4\%$

  $Mg \leq 0.1\%$

  $3 \leq pH \leq 5$

Mineral matter ≥ 95%

- The composition includes, with the contents expressed as mass percentages:

39% ≤ K ≤ 43%

8% ≤ Na ≤ 10%

48% ≤ Cl ≤ 52%

Ca ≤ 0.3%

Mg ≤ 0.01%

3.5 ≤ pH ≤ 4.5

Mineral matter ≥ 97%

The invention also concerns a method for generating the aforementioned dairy origin salt which is characterized essentially in that it includes at the least the steps of:

- generating a concentrated solution of monovalent brine minerals obtained by the electrodialysis of lactoserum, and

- the generation of a dairy origin salt by at least one crystallization step of the concentrated solution of monovalent minerals.

The method of the invention may also include the following optional characteristics considered on their own or in any possible technical combination:

- the generation of the concentrated solution of monovalent brine minerals includes a nanofiltration step to obtain a brine nanofiltration permeate.

- The brine nanofiltration permeate obtained by lactoserum electrodialysis undergoes at least one concentration step to obtain a concentrated nano-filtered brine.

- The brine nanofiltration permeate obtained by lactoserum electrodialysis is concentrated by inverse osmosis.

- The brine nanofiltration permeate undergoes a step to reduce the organic matter content.

- The step to reduce the organic matter content is an absorption step on an adsorbing support forming a deodorization step.

- The generation of a concentrated solution of monovalent brine minerals includes a step to concentrate the solution derived from the reduction step of the organic matter content, followed by a filtration step.

- The concentration step is a vacuum evaporation step.

- The method of the invention includes, in order, the following steps:

- nanofiltration of the brine obtained by lactoserum electrodialysis and the obtaining of a nanofiltration permeate,

- concentration of the nanofiltration permeate by inverse osmosis and the obtaining of a retentate,

- deodorization by adsorption on an absorbing support and obtaining of a solution with a low organic matter content,

- concentration of the solution with a low organic matter content obtained during the previous step, filtration of the solution obtained and obtaining of a concentrated monovalent brine mineral solution.

- The step of generating the dairy origin salt includes at least one forced circulation crystallization step of the concentrated monovalent brine mineral solution and a drying step.

- The step of generating the dairy origin salt includes a centrifugation step prior to the drying step.

Finally, the invention concerns the use of brine obtained by lactoserum electrodialysis to manufacture a crystallized dairy origin salt as defined previously.

Other characteristics and advantages of the invention will appear clearly from the following description, given for information and which is not limiting in any way, with reference to the attached figures among which:

- figure 1 is a block diagram of the two essential steps of the process of preparing a dairy origin salt,

- figure 2 is a block diagram of the process of the invention in one alternative embodiment,

- figure 3 is a micrograph of dairy origin salt crystals of the invention, and

- figure 4 is a micrograph of the salt crystals marketed under the brand name Lactosalt Optitaste®.

The obtaining of a demineralized lactoserum is a known process used for controlling the serum protein and lactose contents of baby milk.

The process of lactoserum demineralization includes in particular an initial electrodialysis step generating on the one hand a lactose which is lean in mineral matter designed to undergo other demineralization steps, and on the other hand, a co-product, the brine, rich in mineral matter.

Table 1 below shows, as an example, the composition of a brine obtained by the electrodialysis of lactoserum. The brine component contents are expressed as a dry extract percentage:

Component	% Dry extract
Na	9.45
K	27.01
K/Na	2.86
Ca	2.33
Mg	0.37
Cl	31.8
P	3.39
Lactates	5.73
Citrates	11.03
Mineral matter	74.78

Table 1

The Applicant has discovered the possibility of using brine, a coproduct of the lactoserum demineralization process, to produce a potassium-rich dairy-origin salt.

The electrodialysis step is carried out in conformity with known techniques by an alternation of anionic and cationic membranes exposed to an electric field.

With reference to figure 1, the brine is exposed to a first step 1 of preparation of a concentrated solution of monovalent minerals and a second step 2 for the preparation of the salt according to conventional industrial techniques.

According to the invention, the first step 1 of the preparation of a concentrated monovalent mineral solution includes at least one first step of nanofiltration 3 of the brine, followed by at least one first step of concentration 4a, a deodorization step by the retention of organic matter 5 and a second concentration step 4b.

Note that both the nanofiltration step 3, and the deodorization step 5, consist in particular in retaining organic matter. Accordingly, the need, in the method of the invention, to extract the brine from the organic matter to obtain a product with concentrated potassium and sodium, is one and the same as the need to obtain a product free of unpleasant odours, on the understanding that the unpleasant odours come from the presence of organic matter.

More generally, the first step 1 in preparing a concentrated monovalent mineral solution therefore includes an alternation of the steps of concentration and extraction of the organic matter, which steps include or provide for, at least one step of enrichment in monovalent minerals.

The second step 2 of preparing the salt from the concentrated monovalent mineral solution of the first step 1 comprises a first crystallization step 6 followed by a centrifugation step 7 and a drying step 8. This second salt preparation step 2 is a way of obtaining a crystallized dairy origin salt similar in texture to a conventional table salt but having a potassium-rich content and a low sodium content, as will be detailed in the following.

According to one preferable alternative of the invention and with reference to figure 2, the brine nanofiltration step 3, obtained by the lactoserum electrodialysis is obtained by means of membranes having a threshold of around 300 Da for an applied pressure included between 20 and 25 bar.

Nanofiltration 3 offers the means of separating the monovalent minerals from the other components of the brine, and in particular from the organic matter and the phosphor. In addition to separating the monovalent minerals, this nanofiltration step 3 of the brine obtained by lactoserum electrodialysis avoids the forming of a unusable paste during subsequent crystallization.

The nanofiltration permeate 10 is thus enriched with monovalent minerals having a low organic matter and bivalent mineral content, which have passed through the nanofiltration retentate 9. The permeate 10 of the nanofiltration 3 has a dry extract percentage of around 1%.

This permeate 10 then goes through a step of inverse osmosis concentration 11 at a pressure included between 25 and 30 bar.

The permeate 12 resulting from the inverse osmosis step 11 comprises essentially water and traces of minerals. The retentate 13 used in the continuation of the process is concentrated in monovalent minerals and has a dry extract percentage of approximately 5%. Furthermore, inverse osmosis has a lower energy concentration than other concentration techniques, such as for instance, concentration on an evaporator.

The retentate 13 of the inverse osmosis step 11 is then subjected to a step of adsorption on resin 14 whereby the remaining organic matter is eliminated. The resins used are deodorization adsorbing resins which offer a certain selectivity regarding the eliminated organic molecules. In particular, these resins are capable of eliminating the volatile fatty acids mainly responsible for the unpleasant odours. The solution obtained has a dry extract percentage of approximately 5%. As an alternative, instead of resins, it is possible to use activated carbon or an active support all of which are grouped together under the name of adsorbing support.

The nano-osmosed and deodorized brine produced in deodorization step 14 then concentrated by vacuum filtration 15 in a falling flow vacuum evaporator. This step increases the dry extract content and avoids the overconsumption of energy during the subsequent crystallization step. The dry extract content at the output of the concentration step is approximately 15%.

Advantageously, the concentration step 15 takes place after the adsorption step on the resin 14 in order to benefit in this last step of soluble dry matter and avoid the fouling of the resins and the alteration of the deodorization capabilities.

The nano-osmosed brine, deodorized and concentrated, is then put through a filtration step 16 to eliminate the calcium phosphate compounds coming from nanofiltration and which could have been formed during the previous concentration step 15 by precipitation at a dry extract content of around 15%.

The deodorized, concentrated and filtered nano-osmosed brine from the filtration step is therefore the monovalent mineral concentrated solution 1. This solution allows the salt to be prepared according to conventional techniques.

For this purpose, and again with reference to figure 2, the solution which is concentrated in monovalent minerals is exposed to the crystallization step 6 by means of a forced flow crystallizer. This step generates the forming of KCl and NaCl crystals by considerably increasing the dry extract content which reaches 67 to 70%. During this step, the saline concentration of the nano-osmosed brine increases up to the solubility limits of the salts.

The nano-osmosed, deodorized, concentrated, filtered and crystallized brine is then centrifuged during a centrifugation step 7 which separates the liquid from the solid phases. This phase is required in particular because the liquid phase can contain traces of organic matter. The extract dry content increases considerably during this step, reaching approximately 95%.

The product is then dried in a drying step on a fluidized bed 8. This step produces a crystallized dairy origin salt whose dry extract content is near 99%. Furthermore, the salt has a texture comparable to a conventional sodium salt, without generating any problems of lumping.

Another known drying technique consists in drying by atomization. However this technique leads to excessive energy consumption.

Drying on a fluidized bed 8 can only be implemented on a product with a high dry extract content. But the method of the invention, through the sequence of previously defined steps, makes it possible to prepare a product with a high dry extract content, allowing drying on a

fluidized bed to obtain a product having a crystallized texture and which can be handled without any lumping phenomena.

Table 2 gives the composition of the dairy origin salt of the invention for three different dry extract contents after crystallization, compared to the composition of the Lactosalt Optitaste® measured by the Applicant.

We see a K/Na ratio varying from 4.19 to 5 which is substantially greater than the K/Na ratio of 2.86 of the brine after electrodialysis (Table 1).

This substantial increase of this ratio during the method of the invention allows the potassium content to increase to the detriment of the sodium content, while preserving a sodium rate which is not negligible to prevent the appearance of a metallic taste caused by too high a potassium content.

In addition, the dairy origin salt of the invention is almost free of organic matter because it has a mineral matter content included between 97.29 and 98.5.

Dairy origin salt of the invention				
Component	67% dry extract on outlet from crystallization	70% dry extract on outlet from crystallization	73% dry extract on outlet from crystallization	Lactosalt Optitaste®
Na	9.92	8.50	8.28	9.55
K	41.6	40.2	41.4	33.96
K/Na	4.19	4.73	5	3.55
Ca	0.116	0.29	0.03	0
Mg	0	0	0	0
Cl	49.85	50.3	50.02	45.1
Ph	4.15	4.74	3.93	6.25
Mineral matter	97.69	97.29	98.5	86.65

Table 2

Microscopic analyses and particle size studies were carried out on the dairy origin salt of the invention. Your

We refer to figure 3 illustrating the salt crystals of the invention. Two crystal diameters have been measured in this figure: a first diameter of 161 micrometers and a second diameter of 306 micrometers with the size of these crystals being representative of the crystals forming the salt of the invention.

These results are confirmed by particle size analyses made by the GEA Kestner Company performing crystallization tests for three dry extract contents as mentioned in Table 2.

The results are as follows:

For a salt containing 67% dry extract after crystallization, more than 50% of the crystals have an average diameter included between 150 and 220 micrometers.

For a salt comprising 70% dry extract after crystallization, more than 50% of the crystals have an average diameter included between 155 and 175 micrometers.

For a salt comprising 67% dry extract after crystallization, more than 50% of the crystals have an average diameter included between 155 and 210 micrometers.

The crystallized salt of the invention permits this salt to reach a texture comparable to that of low sodium salt.

Conversely, as illustrated in figure 4, the size of the Lactosalt Optitaste® crystals is far smaller than 100 micrometers, leading to the texture already mentioned above of the Lactosalt Optitaste® which is closer to that of a powder than of a crystallized salt.

Patentkrav

1. Krystalliseret salt fra mælk, hvis sammensætning har et forhold mellem kaliumindholdet og natriumindholdet på mellem 3 og 6, og hos hvilket mere end 50% af krystallerne har en gennemsnitsdiameter større end 100 mikrometer.

2. Salt fra mælk ifølge krav 1, **kendetegnet ved at** forholdet mellem kaliumindholdet og natriumindholdet er mellem 4 og 6.

3. Salt fra mælk ifølge et hvilket som helst af de foregående krav, **kendetegnet ved at** dets sammensætning omfatter, med indholdene udtrykt som procenter efter vægt:

$35\% \leq K \leq 45\%$
$7\% \leq Na \leq 11\%$
$45\% \leq Cl \leq 55\%$
$Ca \leq 0{,}4\%$
$Mg \leq 0{,}1\%$
$3 \leq pH \leq 5$
Mineralstof $\geq 95\%$

4. Salt fra mælk ifølge et hvilket som helst af de foregående krav, **kendetegnet ved at** dets sammensætning omfatter, med indholdene udtrykt som procenter efter vægt:

$39 \leq K \leq 43\%$
$8\% \leq Na \leq 10\%$
$48\% \leq Cl \leq 52\%$
$Ca \leq 0{,}3\%$
$Mg \leq 0{,}01\%$
$3{,}5 \leq pH \leq 4{,}5$
Mineralstof $\geq 97\%$

5. Fremgangsmåde til fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 1 til 4, **kendetegnet ved at** den mindst omfatter trinnene at;

- fremstille en koncentreret opløsning af monovalente saltvandsmineraler, der er opnået ved elektrodialyse af valle (1), og

- fremstille et salt fra mælk (2) ved mindst et trin med krystallisering (6) af den koncentrerede opløsning af monovalente mineraler.

6. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 5, **kendetegnet ved at** fremstillingen af den koncentrerede opløsning af monovalente saltvandsmineraler omfatter et nanofiltreringstrin (3) som gør det muligt at opnå et nanofiltreringspermeat af saltvandet.

7. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 6, **kendetegnet ved at** nanofiltreringspermeatet af saltvandet opnået ved elektrolyse af valle udsættes for mindst et koncentreringstrin (4a, 4b) på en sådan måde, at der opnås et koncentreret nanofiltreret saltvand.

8. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 7, **kendetegnet ved at** nanofiltreringspermeatet af saltvandet opnået ved elektrolyse af valle koncentreres ved omvendt osmose (11).

9. Fremgangsmåde til fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 6 til 8, **kendetegnet ved at** nanofiltreringspermeatet af saltvandet udsættes for et trin til at reducere indholdet af organiske materialer (5, 14).

10. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 9, **kendetegnet ved at** trinnet til at reducere indholdet af organiske materialer (5, 14) er et adsorptionstrin på en adsorberende bærer (14) som udgør et trin til desodorisering.

11. Fremgangsmåde til fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 9 og 10, **kendetegnet ved at** fremstillingen af en koncentreret opløsning af monovalente saltvandsmineraler omfatter et trin til at koncentrere (15) den opløsning, som kommer fra trinnet til at reducere indholdet af organiske materialer (5, 14), efterfulgt af et filtreringstrin.

12. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 11, **kendetegnet ved at** trinnet til koncentrering (15) er et trin til fordampning i et vakuum (15).

13. Fremgangsmåde til fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 5 til 12, **kendetegnet ved at** den omfatter de følgende successive trin:

- nanofiltrering (3) af saltvandet, der er opnået ved elektrolyse af valle, og opnåelse af et nanofiltreringspermeat,
- koncentrering (4) af nanofiltreringspermeatet ved omvendt osmose (11) og opnåelse af et retentat,
- desodorisering ved adsorption på en adsorberende bærer (14) og opnåelse af en opløsning med et lavt indhold af organiske materialer,
- koncentrering (15) af den i det foregående trin opnåede opløsning med et lavt indhold af organisk materiale, filtrering (16) af den opnåede opløsning og opnåelse af en koncentreret opløsning af monovalente saltvandsmineraler.

14. Fremgangsmåde til fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 5 til 12, **kendetegnet ved at** trinnet til fremstilling af saltet fra mælk omfatter mindst et krystalliseringstrin (6) med tvungen cirkulation af den koncentrerede opløsning af monovalente saltvandsmineraler, og et tørringstrin (8).

15. Fremgangsmåde til fremstilling af et salt fra mælk ifølge krav 14, **kendetegnet ved at** trinnet til fremstilling af saltet fra mælk omfatter et centrifugeringstrin (7) forud for tørringstrinnet (8).

16. Anvendelse af et saltvand opnået ved elektrolyse af valle ved fremstilling af et salt fra mælk ifølge et hvilket som helst af kravene 1 til 4.

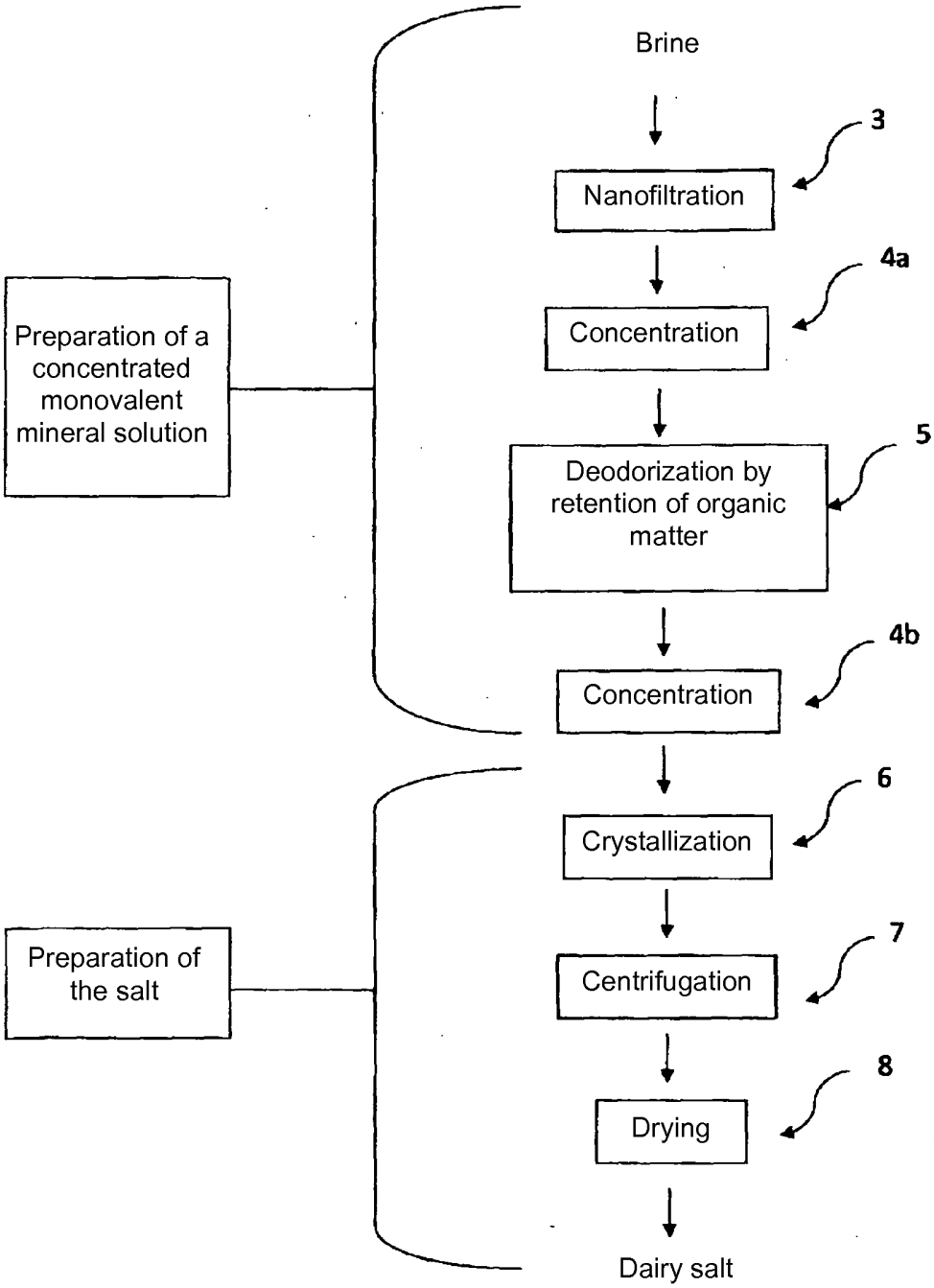
Brine
3
Nanofiltration
4a
Concentration
Preparation of a concentrated monovalent mineral solution
5
Deodorization by retention of organic matter
4b
Concentration
6
Crystallization
Preparation of the salt
7
Centrifugation
8
Drying
Dairy salt

FIGURE 1

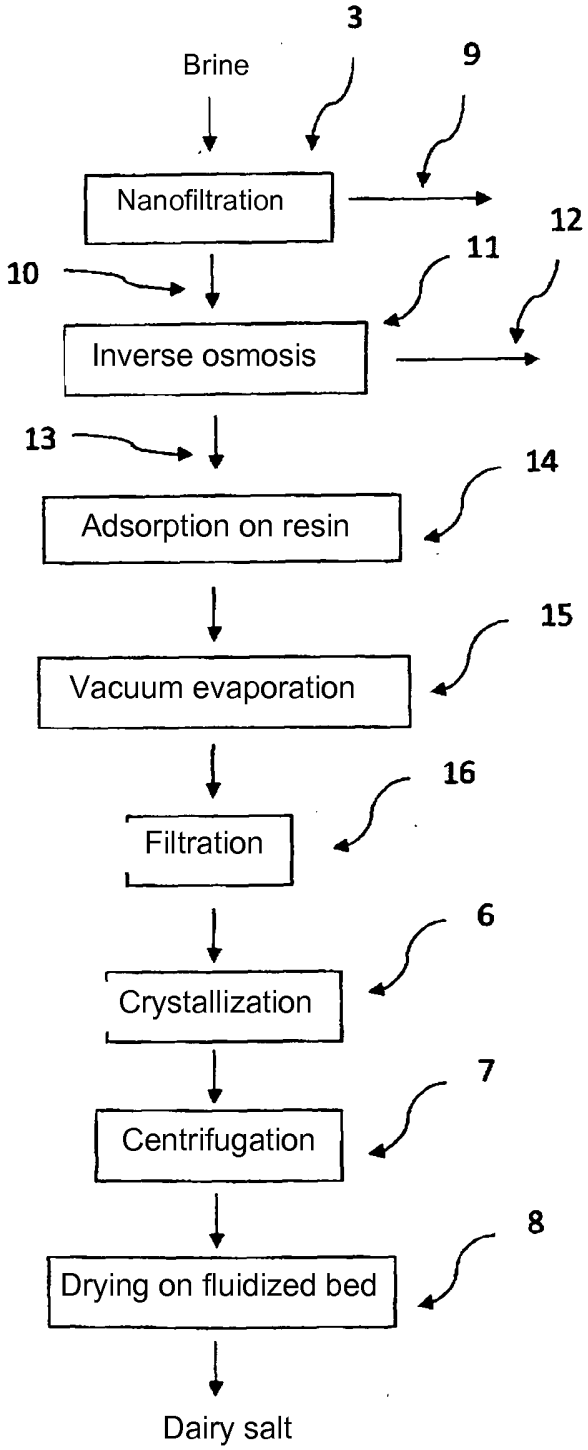
3
Brine
9
Nanofiltration
12
11
10
Inverse osmosis
13
14
Adsorption on resin
15
Vacuum evaporation
16
Filtration
6
Crystallization
7
Centrifugation
8
Drying on fluidized bed
Dairy salt

FIGURE 2

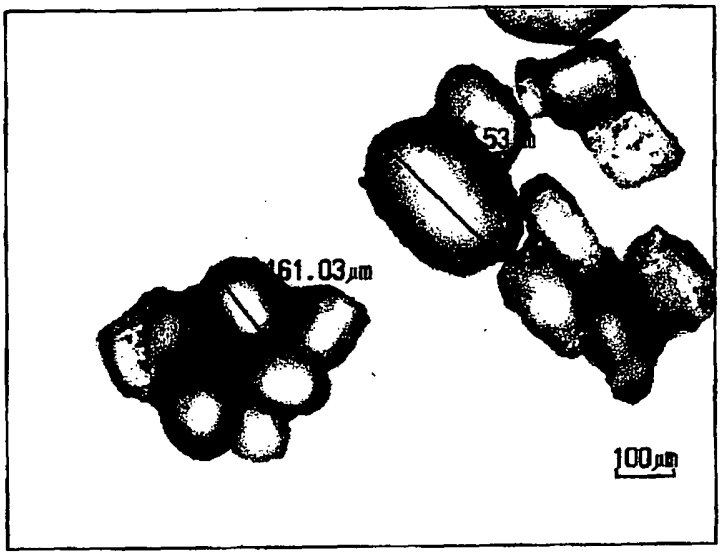

FIGURE 3

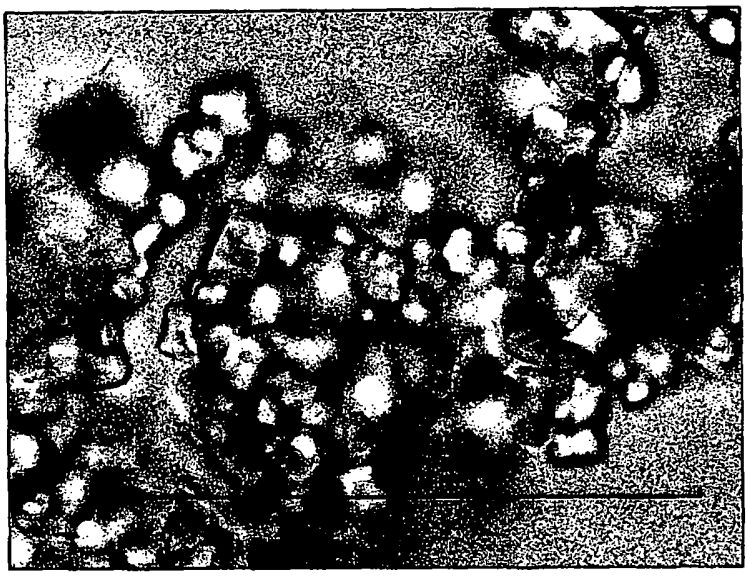

FIGURE 4