

PATENT SPECIFICATION

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(54) PROCESS FOR PREPARING A CONDENSED MILK

(71) We SOCIÉTÉ DES PRODUITS NESTLÉ S.A., of Vevey, Switzerland, a Swiss body corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a process for the production of a vegetable-based condensed milk.

Various kinds of vegetable-based substitutes for the milk of certain mammals, such as the cow, have been known for some considerable time. Thus, it is known that milk substitutes of this type can be obtained from vegetables, such as brussel sprouts, or from coconuts or, above all, from seeds of *leguminosae*, especially soya. In every case, the object is to obtain a colloidal solution of which the composition, appearance and nutritive value are reminiscent of those of an animal milk. This object is achieved by various processes of extraction, purification and titration which hardly contain any secrets. It is also known that it is often possible to produce from these milks, possible at the expense of a slight adjustment in their composition, all kinds of products which are similar to the various standard dairy products, such as cheese and yoghurt.

However, there is a significant gap in this group of known processes and products insofar as vegetable-based condensed milk, more especially a product which will keep for more than a year, is virtually unknown. Although there is a process for producing a condensed milk from almonds, the starting material in question is far too expensive to ever be considered for the industrial production of a competitive substitute for cow's milk. In fact, taking the case of soya, one of the most interesting products both from the economic and from the nutritive point of view, it is noticeable that, although experts may talk of evaporated milks, they never talk

in terms of condensed milks, and that the dry matter content of these evaporated milks does not exceed about 25%.

Despite the fact that known works in the enzymatic modification of proteins have enabled progress to be made in efforts to control the tendency of vegetable proteins, especially soya, to gel in concentrated solutions, there has as yet been no solution to the problem of producing vegetable-based condensed milk. This is because the problem is serious. To realise its seriousness, it is sufficient to remember that a standard condensed cow's milk has a dry matter content of the order of 75% by weight, of which approximately 45% is sugar, which gives approximately 30 parts of milk solids diluted in an aqueous sugar solution containing 45 parts of sugar to about 25 parts of water.

The present invention is the outcome of the search for a solution to this problem.

This present invention provides a process for the production of a vegetable-based condensed milk which comprises subjecting a vegetable protein in the form of an aqueous solution or suspension to partial enzymatic hydrolysis and preparing a mixture containing 4 to 9 parts by weight of fat, 8 to 18 parts by weight of the treated protein, 0 to 14 parts by weight of lactose, 40 to 55 parts by weight of sucrose and 25 to 32 parts by weight of water.

The product obtained by the process according to the invention has a pleasant taste. It is stable. It does not gel, even after prolonged storage.

The process according to the invention may be carried out with all kinds of starting materials rich in vegetable proteins, such as the seeds of *Leguminosa*, especially soya, or microorganisms cultivated for their richness in proteins, especially yeasts.

A condensed milk based on soya proteins may be produced for example from defatted flour, a protein concentrate or a protein iso-

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late. In cases where a defatted soya flour is used as starting material, it may be subjected to an acid (pH-value below 3) or neutral (pH-value above 6.5) or even slightly alkaline extraction process. A solid residue may be separated from the extract, for example by filtration or centrifuging. The proteins present in the supernatant phase may be purified either by ultra-filtration or by isoelectric precipitation, separation and washing for example.

The enzymatic hydrolysis of the vegetable protein is carried out in aqueous solution or suspension. It may be carried out at a temperature of from 25°C to 65°C over a period ranging from 1 to 24 hours and at a pH-value of from 2 to 9, depending on the enzyme. It may be carried out either in batches or continuously in columns containing the enzymes fixed by a support. The enzyme is preferable selected from enzymes of microbial origin, especially bacterial enzymes.

On completion of the enzymatic treatment, the solution or suspension of proteins, obtained from soya or from yeasts for example, may be subjected to an acetylation process intended to increase the whitening power of the end product. This treatment may be carried out over a period of from 20 to 120 minutes at a temperature of from 0 to 30°C and at a pH-value of 6.5 to 10. Acetic anhydride may be used as the acetylating agent in a quantity of 1 part to 10 parts by weight of protein for example. The acetylation treatment may optionally be followed by a purification treatment in the form of isoelectric precipitation and by washing.

The solution or suspension of optionally acetylated proteins may be either concentrated or dried before mixing with the other ingredients included in the composition of the end product. Generally, it is preferred to dry the solution, for example by freeze-drying or by spray drying. The various ingredients may then be mixed in the required proportions. The fat, of which there are 4 to 9 parts in this mixture, may be soya oil, a hydrogenated vegetable fat or any other fat or mixture of vegetable and animal fats with the required physical and nutritional properties. Lactose will be added in cases where it is necessary to comply with formulations requiring, for example, a given percentage of non-fat solids different from sucrose in the end product. It is also possible, if necessary to add salts of the kind commonly encountered in formulations for condensed milk, such as calcium carbonate or a phosphate for example, in a proportion of from 0 to 3% by weight, based on the end product.

Once the various ingredients have been thoroughly mixed, the vegetable-based condensed milk obtained may be pasteurised and homogenised in order further to improve its texture and stability. In this connection, if

lactose has been added to the formulation, it is advisable to crystallise it by conventional methods.

However, it has been found that, by using certain enzymes and, in particular, when it is absolutely essential to inactivate the enzyme by heat treatment, the product obtained may have an excessively high viscosity and sometimes a bitter taste.

Attempts have also been made to overcome this phenomenon.

It has been found that, in order to remedy this possible deficiency, the proteins may be subjected to partial precipitation in suspension and the precipitated fraction eliminated after enzymatic hydrolysis.

In order to obtain this partial precipitation, it is possible either to add a calcium salt, such as CaCl_2 , for example, to the suspension before or after hydrolysis in a concentration of from 0.001 to 0.1M, or to reduce the pH-value of the hydrolysate to a value of from 4.5 to 7, preferably from 5.5 to 6, or even to combine these two techniques. The precipitated proteins may be eliminated by filtration or centrifuging for example. In order to eliminate the possible bitterness mentioned above, attributable to peptides of low molecular weight, the product obtained after hydrolysis may be subjected, for example, to ultrafiltration following removal of the precipitated proteins.

Finally, it has been found that the stability in hot coffee of the vegetable-based condensed milk according to the invention may be increased by adding a basic salt to the mixture. It is preferred to add sodium or potassium bicarbonate in a quantity of 0.1 to 1% by weight, or sodium or potassium carbonate in a quantity of from 0.05 to 1% by weight. It is also possible to use other basic salts, such as Na_2HPO_4 , for example.

In addition, although the fundamental object of the process according to the invention is to produce a purely vegetable condensed milk, it is also possible to replace at least some of the ingredients entering into its composition by milk powder.

The invention is illustrated by the following Examples, in which the parts and percentages quoted are by weight.

EXAMPLE 1

1.5 kg of defatted soya flour are extracted for 45 minutes at ambient temperature in 9 litres of water of which the pH-value has been lowered to 2.6 by the addition of phosphoric acid. After the removal of a solid residue by centrifuging for 15 minutes at 4000 G, the supernatant phase is collected and sodium hydroxide is added to it until its pH-value reaches 4.5. The proteins which have precipitated are collected by centrifuging and are washed three times with water at pH 4.5. They are then dispersed in 9 litres of water of which the pH is adjusted to 7.2 by

the addition of sodium hydroxide. 0.9 g of mixture of proteolytic enzymes of *Streptomyces griseus* (Pronase E, a product of Merck AG, Germany) having an activity of 70 Kunitz units per mg are then added, followed by incubation at 37°C for 4 hours during which the pH is kept at 7.2. The enzymes are inactivated by heating for 20 minutes at 95°C. The solution is frozen and freeze-dried.

90 g of hydrogenated vegetable fat, 100 g of the protein obtained in the manner described above, 500 g of sucrose and 310 g of water are thoroughly mixed. The mixture is homogenised in turbine homogeniser and then introduced into cans which are sterilised for 20 minutes at 100°C.

After storage for 3 months at 20°C and at 30°C, the product has not gelled. It has a pleasant, slightly fruity taste subtly reminiscent of nuts without any hint of bitterness.

EXAMPLE 2

20 kg of defatted soya flour are extracted for 30 minutes at ambient temperature in 120 kg of water of which the pH-value has been lowered to 2.7 by the addition of phosphoric acid. After centrifuging for 20 minutes at 3500 G, the supernatant phase is collected. The pH of the supernatant phase is adjusted to 4.5 by the addition of sodium hydroxide. 85% of the proteins precipitate. They are collected by centrifuging, suspended in 120 kg of water and the pH-value adjusted to pH 7. They are then hydrolysed for 4 hours at 37°C with 30 g of the mixture of enzymes mentioned in Example 1. Hydrolysis is terminated by heating to 100°C. The solution is cooled and dried by spray drying.

15.1% of the proteins obtained, 6.9% of lactose, 7% of hydrogenated soya fat, 43% of sucrose and 28% of water are mixed. 33 kg of vegetable condensed milk with a viscosity of 5280 centipoises (cP) are obtained. After storage for 3 months at 20°C, the viscosity has risen to only 5400 cP and, after 1 month at 37°C, it has only risen to 7200 cP. The product is stable in hot coffee. It has a neutral, if anything slightly fruity taste.

EXAMPLE 3

0.25 kg of defatted soya flour are extracted for 30 minutes at ambient temperature in 2.25 litres of water of which the pH-value has been lowered to 2.7 by the addition of phosphoric acid. The extract is centrifuged at 3000 G for 20 minutes at ambient temperature. The pH-value of the supernatant phase is adjusted to 4.9 by the addition of sodium hydroxide. The precipitate is collected by centrifuging and suspended in 2.5 litres of demineralised water. The suspension is neutralised to pH 7.2 by the addition of sodium hydroxide, followed by the addition of 0.69 g, which is equivalent to 8000 ppm of the dry matter of the solution, of an enzyme of *Bacillus cereus* in powder form having an activity of 1000 units according Keay and Wildi per mg. The enzyme is then incubated at 40°C for 4 hours during which the pH-value is kept between 7 and 7.2. The enzyme is then inactivated by heating. The solution is frozen and freeze-dried. 83 g of hydrolysate are obtained and completely used for the production of a vegetable condensed milk. To this end, 12%, based on the total weight, of hydrolysate, 7% of hydrogenated soya fat, 40% of sucrose, 14% of lactose and 28% of water are thoroughly mixed. A vegetable condensed milk with a viscosity of 2800 cP, as measured with a Brookfield viscosimeter, is obtained.

EXAMPLE 4

A hydrolysate of soya proteins is prepared in the same way as described in Example 3, except that 6000 ppm instead of 8000 ppm of the enzyme in question are used. A vegetable condensed milk is prepared by thoroughly mixing 13%, based on the total weight, of the hydrolysate obtained, 9% of hydrogenated soya fat, 49% of sucrose and 29% of water. The product obtained has a viscosity of 4800 cP, as measured with a Brookfield viscosimeter.

EXAMPLE 5

Single-cell yeast proteins, of which the nucleic acid content has been reduced by alkaline extraction, are suspended in water in a quantity of 1 part of proteins to 20 parts of water, and the pH of the suspension is adjusted to 7.2 by the addition of NaOH. After washing to eliminate the salts, the suspension is heated to 40°C. 0.01 part of the proteolytic enzyme of *Bacillus cereus* mentioned in Example 3 is then added. The temperature is kept at 40°C and the pH-value at 7.2 for 4 hours with constant stirring. The enzyme is inactivated and the troublesome aromas are eliminated by boiling for 20 minutes at ambient pressure. This is followed by centrifuging. The supernatant phase obtained is concentrated and dried.

500 g of the protein material obtained are mixed with 380 g of lactose, 280 g of butter oil, 1720 g of sucrose and 1120 g of water. 4 kg of condensed milk are obtained and are then pasteurised in cans for 5 minutes at 75°C. The product has a viscosity of 9400 cP after storage for 1 month at 25°C.

EXAMPLE 6

A protein hydrolysate obtained in the manner described in Example 3 is dispersed in water in a quantity of 10 parts to 100 parts of water. The pH-value is adjusted to 7.5 with NaOH. The suspension is cooled to 4°C in an ice bath. 1 part of acetic anhydride is added and the mixture left to react at 4°C for 90 minutes during which the pH-value is kept constant. The suspension is dried by freeze-drying.

A condensed milk is prepared with the

hydrolysate thus treated in the manner described in Example 3. The product thus obtained has a distinctly greater whitening power than the product obtained in Example 3.

EXAMPLE 7

5 kg of defatted soya flour are extracted with 435 kg of water for 1 hour while stirring, the pH-value being adjusted to 7 with NaOH. After centrifuging, the supernatant phase is acidified to pH 4.5 with concentrated phosphoric acid. The protein precipitate obtained is recovered by centrifuging. It is redispersed in water and the pH-value adjusted to 7.2 with NaOH. The solution thus obtained is dried by spray drying. A protein with a nitrogen content of around 15%, equivalent to a purity of 94%, is obtained.

1 kg of this protein is dispersed in 14 kg of water. The pH is adjusted to 7.5 by the addition of KOH. The suspension is heated to 60°C on a water bath. 20 g of proteolytic enzyme of *Bacillus subtilis* (Bakterien-proteinase N, a product of Rohm AG, Germany) having an activity of 140 Kunitz units per mg are then added. The mixture is stirred for a period of 24 hours during which the pH is periodically adjusted to 7.5 with KOH. The solution is then directly dried by freeze-drying.

100g of the hydrolysate obtained are thoroughly mixed with 159 g of lactose, 82 g of hydrogenated soya fat, 505 g of sucrose and 1500 g of water. The mixture is homogenised. It is concentrated by evaporation to half its weight. It is then pasteurised by heating at 80°C, followed by cooling in a water bath. The produce has a viscosity of 3600 cP at 25°C. It has not gelled after storage for 1 month at 25°C.

EXAMPLE 8

30 g of protein prepared in the same way as described in Example 7 are dispersed in 270 g of tap water. The pH is adjusted to 3 with HCl. After heating to 40°C, 0.15 g of pepsin (produced by Fluka AG, Switzerland) having an activity of 1/300 units, according to the Manual of the USA National Formulary, 11th Edition, are added. After 3 hours, another 0.15 g of pepsin are added and left to act for another 5 hours during which the pH is kept at pH 3 with HCl and the temperature at 40°C. Hydrolysis is terminated by adjusting the pH to 7.5 with NaOH.

57 g of water, 111 g of sugar, 31 g of lactose and 18 g of hydrogenated soya fat are added to the mass obtained. The produce is then mixed and homogenised. It is concentrated to half its weight by evaporation *in vacuo*. A condensed milk which does not gel and which has a viscosity of 22,000 cP is obtained.

EXAMPLE 9

10 kg of Promine R ("Promine" is a Regis-

tered Trade Mark), which is a commercial soya isolate produced by the Central Soya Corporation of the United States of America) are dispersed in 90 kg of water while adding 135 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. The pH is adjusted to 7 with NaOH. After heating 60°C, 200 g of pancreatin (produced by Fluka AG, Switzerland) are added while stirring. The enzyme is left to incubate for 5 hours during which the pH-value is kept at 7 and the temperature at 60°C. The enzyme is inactivated by heating for 10 minutes to boiling temperature and is then cooled to 20°C. After centrifuging, 79 kg of supernatant phase are collected.

36.5 kg of sucrose, 11.1 kg of lactose, 5.9 kg of melted soya fat and the 70 kg of lactose, 5.9 kg of melted soya fat and the 79 kg of hydrolysate obtained above are mixed at 40°C. The mixture is thoroughly mixed. It is then pasteurised for 1 minute at 80°C in a scraped-surface heat exchanger. It is then concentrated by evaporation *in vacuo* until its dry matter content reaches 72%. It is then cooled and inoculated with lactose. 86 kg of condensed milk with a viscosity of 7200 cP are obtained.

EXAMPLE 10

A hydrolysate is prepared in the same way as described in Example 9, except that the calcium salt is not added. After the enzyme has been inactivated, 25 kg of the suspension are removed. Its pH is lowered to 5.5 with HCl. The suspension is then centrifuged in an Alfa-Lavals ("Alfa-Laval" is a Registered Trade Mark) plate centrifuge and 20 kg of supernatant phase are collected.

1.9 kg of lactose, 6.5 kg of sucrose, 1.06 kg of hydrogenated soya fat and the 20 kg of supernatant phase, of which the pH is adjusted to 7 with NaOH, are mixed at 40°C. The mixture is pasteurised and concentrated in the same way as described in Example 9. It is introduced into cans and sterilised by immersing the cans for 30 minutes in water heated to 80°C. A condensed milk with a viscosity of 22,000 cP is obtained.

EXAMPLE 11

A hydrolysate is prepared from 1.5 kg of soya isolate (Promine R) in the same way and using the same proportions as described in Example 9. After centrifuging, the supernatant phase is diluted to give 25 kg of dilute hydrolysate. The dilute hydrolysate is then filtered in a Seitz ("Seitz" is a Registered Trade Mark) filter press equipped with Koth 7 filters. 20 kg of dilute, filtered hydrolysate are collected and subjected to ultrafiltration in a DDS (De Danske Sukkerfabrikker) pilot module equipped with DDS 800 membranes. 15 kg of permeate are thus eliminated. The concentrate is washed while adding 5 kg of distilled water and continuing the ultrafiltration treatment. Another 5 kg of permeate are eliminated. 6.3 kg of purified

protein suspension are collected.

133 k of lactose, 430 g of sucrose, 70 g of hydrogenated soya fat, 607 g of water and 760 g of the above protein suspension are mixed at 40°C. The mixture is concentrated by evaporation *in vacuo* and then heated to 80°C, followed by cooling. 1 kg of condensed milk with a pleasant taste and a viscosity of 9200 cP is obtained.

EXAMPLE 12

A condensed milk is prepared in the same way as described in Example 11 except that 0.5% of NaHCO₃ is added to the mixture. The product obtained shows no trace of precipitation when used for sweetening and whitening coffee at 80°C.

EXAMPLE 13

A condensed milk is prepared in the same way as described in Example 11 except that, in the recipe of the mixture 230 g of the suspension of vegetable proteins and 40 g of lactose are replaced by 70 g of skimmed milk powder dispersed in 200 g of water. The product obtained has a greater whitening power than the condensed milk of Example 11.

WHAT WE CLAIM IS:

1. A process for the production of a vegetable-based condensed milk, which comprises subjecting a vegetable protein in the form of an aqueous solution or suspension to a partial enzymatic hydrolysis and preparing a mixture containing 4 to 9 parts by weight of fat, 8 to 18 parts by weight of the treated protein, 0 to 14 parts by weight of lactose, 40 to 44 parts by weight of sucrose and 25 to 32 parts by weight of water.

2. A process as claimed in Claim 1, wherein the protein is subjected to an acetylation treatment before the mixture is prepared.

3. A process as claimed in Claim 1 or 2, wherein the mixture is pasteurised and homogenised.

4. A process as claimed in any of Claims 1 to 3, wherein the enzyme is a microbial protease.

5. A process as claimed in any of Claims 1 to 4, wherein the enzymatic treatment is carried out over a period of from 1 to 24 hours at a temperature in the range from 25 to 65°C.

6. A process as claimed in any of Claims 1 to 5, wherein the enzymatic treatment is carried out at a pH-value of from 2 to 9.

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7. A process as claimed in any of Claims 1 to 6, wherein the protein is a protein of *Leguminosae* or a yeast protein.

8. A process as claimed in Claim 7, wherein the protein is a soya protein.

9. A process as claimed in any of Claims 1 to 8, wherein the proteins are subjected to partial precipitation in suspension and the precipitated fraction is eliminated after the enzymatic hydrolysis step.

10. A process as claimed in Claim 9, wherein, to obtain partial precipitation, a calcium salt is added to the suspension in a concentration of from 0.001 to 0.1 M.

11. A process as claimed in Claim 9, wherein to obtain partial precipitation, the pH-value of the hydrolysed suspension is lowered to a value of from 4.5 to 7.

12. A process as claimed in Claim 11, wherein the pH-value is lowered to a value of from pH 5.5 to pH 6.

13. A process as claimed in any Claims 1 to 12, wherein a basic salt is added to the mixture.

14. A process as claimed in Claim 13, wherein the basic salt is added in a quantity of from 0.05 to 1% by weight.

15. A process as claimed in Claim 14, wherein the basic salt is sodium or potassium carbonate or bicarbonate.

16. A process as claimed in Claim 1 for the production of a vegetable-based condensed milk substantially as described with particular reference to any of Examples 1 to 8.

17. A process as claimed in Claim 1 for the production of a vegetable-based condensed milk substantially as described with particular reference to any of Examples 9 to 13.

18. A vegetable-based condensed milk when produced by a process as claimed in any of Claims 1 to 8 or 16.

19. A vegetable-based condensed milk when produced by a process as claimed in any of Claims 9 to 15 or 17.

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EXAMPLE 12

A condensed milk is prepared in the same way as described in Example 11 except that 0.5% of NaHCO₃ is added to the mixture. The product obtained shows no trace of precipitation when used for sweetening and whitening coffee at 80°C.

EXAMPLE 13

A condensed milk is prepared in the same way as described in Example 11 except that, in the recipe of the mixture 230 g of the suspension of vegetable proteins and 40 g of lactose are replaced by 70 g of skimmed milk powder dispersed in 200 g of water. The product obtained has a greater whitening power than the condensed milk of Example 11.

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1. A process for the production of a vegetable-based condensed milk, which comprises subjecting a vegetable protein in the form of an aqueous solution or suspension to a partial enzymatic hydrolysis and preparing a mixture containing 4 to 9 parts by weight of fat, 8 to 18 parts by weight of the treated protein, 0 to 14 parts by weight of lactose, 40 to 44 parts by weight of sucrose and 25 to 32 parts by weight of water.
2. A process as claimed in Claim 1, wherein the protein is subjected to an acetylation treatment before the mixture is prepared.
3. A process as claimed in Claim 1 or 2, wherein the mixture is pasteurised and homogenised.
4. A process as claimed in any of Claims 1 to 3, wherein the enzyme is a microbial protease.
5. A process as claimed in any of Claims 1 to 4, wherein the enzymatic treatment is carried out over a period of from 1 to 24 hours at a temperature in the range from 25 to 65°C.
6. A process as claimed in any of Claims 1 to 5, wherein the enzymatic treatment is carried

ried out at a pH-value of from 2 to 9.

7. A process as claimed in any of Claims 1 to 6, wherein the protein is a protein of *Leguminosae* or a yeast protein.

8. A process as claimed in Claim 7, wherein the protein is a soya protein.

9. A process as claimed in any of Claims 1 to 8, wherein the proteins are subjected to partial precipitation in suspension and the precipitated fraction is eliminated after the enzymatic hydrolysis step.

10. A process as claimed in Claim 9, wherein, to obtain partial precipitation, a calcium salt is added to the suspension in a concentration of from 0.001 to 0.1 M.

11. A process as claimed in Claim 9, wherein to obtain partial precipitation, the pH-value of the hydrolysed suspension is lowered to a value of from 4.5 to 7.

12. A process as claimed in Claim 11, wherein the pH-value is lowered to a value of from pH 5.5 to pH 6.

13. A process as claimed in any Claims 1 to 12, wherein a basic salt is added to the mixture.

14. A process as claimed in Claim 13, wherein the basic salt is added in a quantity of from 0.05 to 1% by weight.

15. A process as claimed in Claim 14, wherein the basic salt is sodium or potassium carbonate or bicarbonate.

16. A process as claimed in Claim 1 for the production of a vegetable-based condensed milk substantially as described with particular reference to any of Examples 1 to 8.

17. A process as claimed in Claim 1 for the production of a vegetable-based condensed milk substantially as described with particular reference to any of Examples 9 to 13.

18. A vegetable-based condensed milk when produced by a process as claimed in any of Claims 1 to 8 or 16.

19. A vegetable-based condensed milk when produced by a process as claimed in any of Claims 9 to 15 or 17.

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