HIGH-SOLUBILITY CASEIN POWDER AND PREPARATION METHOD THEREFOR

[0001] Priority Claim

[0002] This application claims priority benefit of Chinese Patent Application No. 2024104807808, filed on April 22, 2024, entitled "HIGH-SOLUBILITY CASEIN POWDER AND PREPARATION METHOD THEREFOR".

Technical Field

[0003] The present invention relates to the technical field of dairy product processing, and in particular to a high-solubility casein powder and a preparation method therefor.

Background Art

[0004] Casein is the most abundant protein with the highest content in bovine milk, accounting for about 80% of the total protein content in milk. Casein is composed of four proteins, namely α s1-casein, α s2-casein, β-casein, and κ-casein. The most prominent feature of casein is that, in a milk system, strong interactions occur among the four casein subtypes, which form a stable micellar structure with nanoclusters composed of Ca and P elements in the milk system. At present, there is no universally recognized model for casein micelles. Current research mainly focuses on the subunit model, the Holt model, and the dual-binding model (Horne model). Regardless of the model used for research, the integrity of the micelles has a highly significant impact on the overall properties of the micelles, the stability of the milk system, the application characteristics of a product, and its nutritional properties.

[0005] The solubility of casein or casein powder is particularly affected by the integrity of the casein micelles. The four casein subtypes exhibit distinct differences in their hydrophilic/hydrophobic structures. α s1-casein, α s2-casein, and β-casein, which lack hydrophilic structures, form nanoclusters with Ca and P ions through ionic interactions at the center of the micelle. In contrast, κ-casein is mainly concentrated on the surface of the micelle and possesses a distinct amphiphilic structure, where the hydrophobic portion binds with the nanoclusters via intermolecular forces to maintain the overall micelle structure, and the hydrophilic portion is positioned on the outer surface of the micelle to maintain micelle solubility. Under certain specific circumstances, hydrophobic portion is exposed due to the disruption of the micelle structure, leading to a decrease in casein solubility or even complete precipitation (for example, precipitating casein by lowering the pH value or using rennet during cheese making). Therefore, improving the integrity of casein micelles is crucial for increasing their solubility. The structural stability of casein micelles may be influenced by various factors, such as temperature, heat treatment intensity, pH value, pressure, ion concentration, chelating agents, and enzymes.

[0006] Currently, there are three main methods for preparing casein powder: (1) acid casein, in which the acid casein powder is prepared by adding starter cultures or acids to reduce the pH of the solution below the isoelectric point of casein to precipitate the casein, followed by processes such as heating, water washing, separation, and drying; (2) rennet casein powder, in which the rennet casein powder is prepared by adding rennet until the casein coagulates, followed by processes such as heating, water washing, separation, and drying; and (3) membrane separation method, in which the membrane-method casein micelle powder is prepared by microfiltration/ultrafiltration membrane separation using skim milk as a raw material, followed by processes such as separation, diafiltration (wash-filtration), concentration, and drying. Because the acid method and the enzyme method completely and irreversibly destroy the micelle structure of casein during preparation, resulting in extremely poor

solubility upon reconstitution, they are not discussed in detail herein. Microfiltration membrane separation technology is an efficient, energy-saving, and environmentally friendly non-thermal physical separation technology. It is suitable for separating temperature-sensitive, heat-labile high-value nutrients and can maintain the integrity of casein micelles during separation to a certain extent. However, casein powder obtained by existing membrane separation methods remains mediocre solubility as disclosed, for example, in CN103315128A, and still requires further improvement.

Summary of the Invention

[0007] To address the deficiencies of the prior art, the present invention provides a high-solubility casein powder and a preparation method therefor.

[0008] Specifically, in a first aspect, the present invention provides a method for preparing a casein powder, comprising sequentially subjecting skim milk to microfiltration, ultrafiltration, and drying steps,

[0009] wherein in the microfiltration step, a volume concentration factor is 1 to 5; and

[0010] in the ultrafiltration step, an ultrafiltration membrane having a molecular weight cut-off of 30 kDa to 100 kDa is used, the concentration temperature is 10°C to 20°C, the concentration pressure is 0.1 to 1 mPa, and a total solids content after concentration is 10% to 25%.

[0011] Herein, the volume concentration factor (VCF) refers to the ratio of the total feed volume to the volume of the concentrated liquid.

[0012] In the present invention, microfiltration is first employed to remove most of the whey proteins, followed by ultrafiltration to remove residual whey proteins and other impurities such as miscellaneous proteins and minerals. The concentration process of the present invention is a non-thermal concentration process, thereby preventing damage to the micellar structure of the micellar casein concentrate (MCC). The present invention does not require additional processing aids; by controlling process parameters during separation, casein micelles can be separated from other impurities under mild conditions.

[0013] Herein, the temperature selection during ultrafiltration concentration is critical. If the temperature is too low, β-casein may dissociate from the micelle of the MCC, leading to micellar structural damage, an increase in feed liquid viscosity, thereby reducing production efficiency. If the temperature is too high, microbial growth in the feed liquid may become uncontrolled, increasing microbial risks during production; however, a higher temperature can reduce feed liquid viscosity and improve concentration efficiency. After comprehensive consideration, the concentration temperature selected in the present invention is 10°C to 20°C, which controls microbial proliferation while ensuring the integrity and proportion of casein subtypes within the casein micelles. In a preferred embodiment of the present invention, the concentration temperature is 15°C. The molecular weight cut-off of ultrafiltration membranes spans a wide range, with the molecular weight distribution of ultrafiltration membranes ranging from 3,000 Da to 500 kDa. When the temperature drops to around 10°C, β-casein (β-CN) dissociates from the casein micelles, resulting in incomplete casein micellar structures and consequently poor solubility of the final MCC powder. Since the molecular weights of both β-casein and whey proteins are approximately 20 kDa to 30 kDa, a 30-kDa to 100-kDa ultrafiltration membrane is selected to filter out impurity proteins while retaining micellar casein. In addition, the concentration of the final concentrated feed liquid is also crucial. If the concentration is too low, preparation efficiency is low; if the concentration is too high, it may lead to excessively high viscosity of the feed liquid, poor fluidity, and increased gelation, which may clog the atomizer during subsequent drying.

Therefore, to ensure product quality and production continuity, it is necessary to control the total solids content after concentration within the range of 10% to 25%. Furthermore, with respect to the concentration temperature and total solids content after concentration, an appropriate concentration pressure is required to ensure concentration efficiency and production continuity. That is, the parameters in the present invention are closely interrelated and synergistically act to achieve continuous production and yield casein powder with high solubility.

[0014] In a preferred embodiment of the present invention, in the ultrafiltration step, an ultrafiltration membrane with a molecular weight cut-off of 50 kDa is used; the concentration temperature is 15°C; the concentration pressure is 0.6 MPa; and the total solids content after concentration is 20%.

[0015] In some embodiments of the present invention, in the microfiltration step, the ratio of diafiltration water volume to retentate volume is 2:1 to 6:1; and in the ultrafiltration step, the ratio of diafiltration water volume to total feed volume is 0.5:1 to 3:1.

[0016] Further preferably, in both the microfiltration and the ultrafiltration steps, the diafiltration mode employs inline addition of diafiltration water. Inline addition of diafiltration water of the present application is superior to conventional batchwise addition, as it enables continuous production.

[0017] Further preferably, in the microfiltration step, the ratio of diafiltration water volume to retentate volume is 4.5:1; and

[0018] in the ultrafiltration step, the ratio of diafiltration water volume to total feed volume is 1:1.

[0019] Utilizing an inline diafiltration process with appropriate flow rate ratios in the microfiltration and ultrafiltration steps of the present invention enables continuous production and aids in improving casein separation rate, casein purity, and production efficiency, thereby achieving economic benefits.

[0020] In some embodiments of the present invention, in the microfiltration step, a microfiltration membrane with a pore size of 0.1 to 0.2 micrometers is used; the separation temperature is 40°C to 60°C; and the transmembrane pressure is 0.1 to 0.2 mPa. Preferably, in some embodiments of the present invention, in the microfiltration step, a microfiltration membrane with a pore size of 0.14 micrometers is used.

[0021] Further preferably, in the microfiltration step, a microfiltration membrane with a pore size of 0.14 micrometers is used, the separation temperature is 50°C, the transmembrane pressure is 0.1 to 0.2 mPa, and the volume concentration factor is 3.

[0022] In some embodiments of the present invention, the drying step is performed by using electrostatic spray drying.

[0023] The present invention employs electrostatic spray drying. Compared with conventional spray drying, electrostatic spray drying can significantly reduce the degree of heat exposure of the feed liquid during the drying at a lower moisture content, thereby protecting the structural integrity of the casein micelles and improving the solubility of the MCC powder upon reconstitution.

[0024] In some embodiments of the present invention, in the electrostatic spray drying step, the atomizing nozzle uses a pressure atomizer; an atomization pressure is 0.1 to 2 mPa, an atomization temperature is 20°C to 60°C, a protective gas is nitrogen, an inlet air temperature is 60°C to 90°C, an outlet air temperature is 30°C to 50°C, an electrostatic field voltage is 1 to 20 kV, and a material preheating temperature is 30°C to 60°C.

[0025] Furthermore, in a preferred embodiment of the present invention, the atomization pressure is 0.3 mPa, the atomization temperature is 40°C, the protective gas is nitrogen, the inlet air temperature is 70°C, the outlet air temperature is 40°C, the electrostatic field voltage is 12 kV, and the material preheating temperature is 50°C.

[0026] The skim milk of the present invention is obtained by standardizing raw bovine milk, followed by skimming and pasteurization. The skimming temperature is 45°C to 55°C, preferably 50°C. The pasteurization temperature is 60°C to 90°C, and the pasteurization time is 10 s to 60 s.

[0027] Furthermore, the fat content in the skim milk is 0.01% to 0.1%. It is found in the present invention that the fat content in skim milk has little impact on product solubility, but affects the sustainable duration of membrane filtration process treatment. For production continuity, the fat content in the skim milk is preferably controlled within 0.01% to 0.1% in the present invention.

[0028] In some embodiments of the present invention, the method for preparing the casein powder comprises the following steps:

[0029] (1) Fresh raw bovine milk is subjected to a standardization treatment to obtain standardized raw milk. The raw milk is subjected to a skimming process, wherein the skimming temperature is 45°C to 55°C, and the fat content in the skim milk is 0.01% to 0.1%; the skim milk is subjected to a pasteurization treatment, wherein the pasteurization temperature is 60°C to 90°C, and the pasteurization time is 10 to 60 s; (2) Microfiltration membrane separation step: the pasteurized skim milk enters a microfiltration membrane separation device, wherein the separation temperature is 40°C to 60°C, the transmembrane pressure is 0.1 to 0.2 mPa, the volume concentration factor is 1 to 5, the ratio of diafiltration water volume to retentate volume is 2:1 to 6:1, and the diafiltration is performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation enters an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature is 10°C to 20°C, the concentration pressure is 0.1 to 1 mPa, the diafiltration is performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume is 0.5:1 to 3:1, the total solids content after concentration is 10% to 25%, the protein content is 17% to 19%, and casein accounts for 85% to 97% of the total protein; (4) Drying step: wherein the atomizing nozzle uses a pressure atomizer, the atomization pressure is 0.1 to 2 mPa, the atomization temperature is 20°C to 60°C, the protective gas is nitrogen, the inlet air temperature is 60°C to 90°C, the outlet air temperature is 30°C to 50°C, the electrostatic field voltage is 1 to 20 kV, and the material preheating temperature is 30°C to 60°C.

[0030] By controlling process conditions, the present invention preserves, to the greatest extent possible, the relatively intact the micellar structure of casein throughout the entire process flow from pretreatment to concentration and drying. Consequently, after reconstitution, the MCC powder still exhibits excellent solubility, thereby resolving the core technical problem of poor solubility that restricts MCC powder application.

[0031] In another aspect, the present invention also provides a casein powder prepared by the above-described method.

[0032] The casein powder obtained by the present invention possesses a relatively intact micellar structure and exhibits excellent solubility upon reconstitution, superior to mainstream casein powders currently on the market. The casein powder has a centrifugal sedimentation rate of less than 28%, as determined according to GB 5413.29-2010 and a dispersibility of greater than 46%, as determined according to IDF 87:1979.

[0033] The present invention provides a high-solubility casein powder and a preparation method therefor. By controlling the key process parameters in each production step, the resulting casein powder is ensured, as much as possible, to have a relatively intact micellar structure, so that the casein powder retains excellent solubility after reconstitution to expand its application prospects. Moreover, the preparation method of the present invention boasts high production efficiency, strong continuous

production capability, and commercial cost-effectiveness.

Brief Description of the Drawings

[0034] Fig. 1 is a process flow diagram for preparing casein powder in an example of the present invention.

[0035] Fig. 2 is an SEM image of the casein powder obtained in Example 1 of the present invention.

[0036] Fig. 3 is an SEM image of the MCC powder obtained in Comparative Example 3 using conventional spray drying.

[0037] Fig. 4 is an SEM image of the MCC powder obtained in Comparative Example 4 using freeze drying.

[0038] Fig. 5 is a graph showing static solubility test results of the casein powders obtained in Examples 1 to 7 (from left to right: Examples 1 to 7).

[0039] Fig. 6 is a graph showing static solubility test results of the casein powders obtained in Comparative Examples 1 to 6 (from left to right: Comparative Examples 1 to 6).

[0040] Fig. 7 is a graph showing stability test results of the casein powders obtained in Examples 1 to 3 (from left to right: Examples 1 to 3).

[0041] Fig. 8 is a graph showing stability test results of the casein powders obtained in Examples 4 to 7 (from left to right: Examples 4 to 7).

[0042] Fig. 9 is a graph showing stability test results of the casein powders obtained in Comparative Examples 1 to 3 (from left to right: Comparative Examples 1 to 3).

[0043] Fig. 10 is a graph showing stability test results of the casein powders obtained in Comparative Examples 4 to 6 (from left to right: Comparative Examples 4 to 6).

Detailed Description of the Invention

[0044] To render the objects, technical solutions, and advantages of the present invention clearer, the technical solutions are described in detail below. The described embodiments are only a part, rather than all, of the embodiments of the present invention. Based on the embodiments of the present invention, all other embodiments obtained by a person of ordinary skill in the art without creative effort shall fall within the protection scope of the present invention.

[0045] Unless otherwise specified, technical means or conditions applied herein follow those described in the relevant literature in the art or product manuals. Reagents or instruments without specified manufacturers are conventional products commercially available through standard channels.

[0046] In the following examples, the microfiltration membrane pore size in the microfiltration step is 0.14 micrometers; and

[0047] the ultrafiltration membrane used in the ultrafiltration step has a molecular weight cut-off of 50 kDa.

[0048] Example 1

[0049] This example provided a casein powder with high solubility. The preparation process flow is shown in Fig. 1, and the specific preparation method was as follows:

[0050] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration

membrane separation device, wherein the separation temperature was 50°C, the transmembrane pressure was 0.15 mPa, the volume concentration factor was 3, the ratio of diafiltration water volume to retentate volume was 4.5:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 15°C, the concentration pressure was 0.6 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 1:1, the total solids content after concentration was 20%, the protein content was 18%, and casein accounted for 94% of the total protein; (4) Electrostatic spray drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 0.3 mPa, the atomization temperature was 40°C, the protective gas was nitrogen, the inlet air temperature was 70°C, the outlet air temperature was 40°C, the electrostatic field voltage was 12 kV, and the material preheating temperature was 50°C.

[0051] The casein powder obtained in this example was subjected to scanning electron microscopy (SEM) detection, and the obtained SEM image is shown in Fig. 2. It can be seen that the powder has a uniform particle size distribution, overall between 80 and 100 micrometers, and the particle surfaces exhibit obvious collapse.

[0052] Example 2

[0053] This example provided a casein powder with high solubility. The preparation method was as follows:

[0054] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 45°C, and the fat content in the skim milk was 0.05%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 70°C, and the pasteurization time was 40 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 50°C, the transmembrane pressure was 0.15 mPa, the volume concentration factor was 4, the ratio of diafiltration water volume to retentate volume was 4:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 10°C, the concentration pressure was 0.5 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 2:1, the total solids content after concentration was 20%, the protein content was 18%, and casein accounted for 96% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 0.5 mPa, the atomization temperature was 40°C, the protective gas was nitrogen, the inlet air temperature was 75°C, the outlet air temperature was 40°C, the electrostatic field voltage was 10 kV, and the material preheating temperature was 45°C.

[0055] Example 3

[0056] This example provided a casein powder with high solubility. The preparation method was as follows:

[0057] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2)

Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 50°C, the transmembrane pressure was 0.2 mPa, the volume concentration factor was 3, the ratio of diafiltration water volume to retentate volume was 5:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 20°C, the concentration pressure was 0.4 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 0.8:1, the total solids content after concentration was 20%, the protein content was 19%, and casein accounted for 94% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 0.8 mPa, the atomization temperature was 40°C, the protective gas was nitrogen, the inlet air temperature was 70°C, the outlet air temperature was 40°C, the electrostatic field voltage was 14 kV, and the material preheating temperature was 50°C.

[0058] Example 4

[0059] This example provided a casein powder with high solubility. The preparation method was as follows:

[0060] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 40°C, the transmembrane pressure was 0.13 mPa, the volume concentration factor was 5, the ratio of diafiltration water volume to retentate volume was 4:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 10°C, the concentration pressure was 0.3 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 3:1, the total solids content after concentration was 20%, the protein content was 17%, and casein accounted for 88% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 2 mPa, the atomization temperature was 30°C, the protective gas was nitrogen, the inlet air temperature was 60°C, the outlet air temperature was 40°C, the electrostatic field voltage was 20 kV, and the material preheating temperature was 50°C.

[0061] Example 5

[0062] This example provided a casein powder with high solubility. The preparation method was as follows:

[0063] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 50°C, the transmembrane pressure was 0.1 mPa, the volume concentration factor was 3, the ratio of diafiltration water volume to retentate volume was 6:1, and the diafiltration was performed in an inline addition mode; (3)

Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 14°C, the concentration pressure was 0.1 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 2:1, the total solids content after concentration was 25%, the protein content was 19%, and casein accounted for 85% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 1 mPa, the atomization temperature was 60°C, the protective gas was nitrogen, the inlet air temperature was 70°C, the outlet air temperature was 30°C, the electrostatic field voltage was 10 kV, and the material preheating temperature was 60°C.

[0064] Example 6

[0065] This example provided a casein powder with high solubility. The preparation method was as follows:

[0066] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 60°C, the transmembrane pressure was 0.18 mPa, the volume concentration factor was 1, the ratio of diafiltration water volume to retentate volume was 5:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 20°C, the concentration pressure was 0.9 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total feed volume was 0.5:1, the total solids content after concentration was 14%, the protein content was 19%, and casein accounted for 95% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 0.1 mPa, the atomization temperature was 40°C, the protective gas was nitrogen, the inlet air temperature was 90°C, the outlet air temperature was 50°C, the electrostatic field voltage was 1 kV, and the material preheating temperature was 40°C.

[0067] Example 7

[0068] This example provided a casein powder with high solubility. The preparation method was as follows:

[0069] (1) Fresh raw bovine milk was subjected to a standardization treatment to obtain standardized raw milk. The raw milk was subjected to a skimming process, wherein the skimming temperature was 50°C, and the fat content in the skim milk was 0.06%; the skim milk was subjected to a pasteurization treatment, wherein the pasteurization temperature was 75°C, and the pasteurization time was 30 s; (2) Microfiltration membrane separation step: the pasteurized skim milk entered a microfiltration membrane separation device, wherein the separation temperature was 55°C, the transmembrane pressure was 0.2 mPa, the volume concentration factor was 4, the ratio of diafiltration water volume to retentate volume was 2:1, and the diafiltration was performed in an inline addition mode; (3) Ultrafiltration membrane concentration step: the casein feed liquid obtained after microfiltration membrane separation entered an ultrafiltration membrane device for ultrafiltration concentration, wherein the concentration temperature was 18°C, the concentration pressure was 1 mPa, the diafiltration was performed in an inline addition mode, the ratio of diafiltration water volume to total

feed volume was 2.5:1, the total solids content after concentration was 10%, the protein content was 17%, and casein accounted for 97% of the total protein; (4) Drying step: wherein the atomizing nozzle used a pressure atomizer, the atomization pressure was 0.8 mPa, the atomization temperature was 20°C, the protective gas was nitrogen, the inlet air temperature was 80°C, the outlet air temperature was 50°C, the electrostatic field voltage was 15 kV, and the material preheating temperature was 30°C.

[0070] Comparative Example 1

[0071] This comparative example provided a conventional acid-process casein powder.

[0072] Comparative Example 2

[0073] This comparative example provided a conventional enzyme-process casein powder.

[0074] Comparative Example 3

[0075] This comparative example provided a casein powder, which differed from Example 1 in that step (4) adopted a conventional spray drying process: inlet air temperature 150°C, outlet air temperature 80°C.

[0076] Fig. 3 is the SEM image of the MCC powder obtained by conventional spray drying in this comparative example. The particle size distribution was non-uniform, ranging from 60 to 200 micrometers; the powder particle morphology was different, with some particles having a collapsed surface and some having a smooth surface.

[0077] Comparative Example 4

[0078] This comparative example provided a casein powder, which differed from Example 1 in that step (4) adopted freeze drying, and the freezing temperature was -89°C.

[0079] Fig. 4 is the SEM image of the MCC powder obtained by freeze drying in this comparative example. It can be seen that the appearance of the freeze-dried MCC powder is different from that of the MCC powder obtained by spray drying in the examples of the present invention, and it is in the form of flaky crystals.

[0080] Comparative Example 5

[0081] This comparative example provided a casein powder, which differed from Example 1 in that in step (3), the concentration temperature was 30°C.

[0082] Comparative Example 6

[0083] This comparative example provided a casein powder, which differed from Example 1 in that in step (3), the total solids content after concentration was 40%.

[0084] Performance Tests

[0085] The casein powders obtained in the examples and comparative examples were subjected to performance tests, including:

[0086] 1. Static solubility test

[0087] An equal mass of purified water was placed in a test tube, and an equal mass of the MCC powder was simultaneously poured into the test tube to observe the dissolution of the MCC powder under no external force.

[0088] The results are shown in Fig. 5 and Fig. 6. The samples with excellent solubility spontaneously and rapidly settled downward and dissolved during the settling process, forming a white solution; for the samples with poor solubility, the casein powder floated on the surface, the settling speed was significantly slower, and the liquid color did not change significantly. It can be seen that the static solubility of Examples 1 to 7 is significantly better than that of Comparative Examples 1 to 6, especially better than that of Comparative Examples 1 to 4.

[0089] 2. Stability test

[0090] An equal mass of the MCC powder was dissolved in purified water at 50°C to prepare a casein solution with a mass concentration of 3%. The solution was kept warm and stirred for hydration for 30 min, and then allowed to stand at room temperature for 8 hours to observe the stability.

[0091] The results are shown in Figs. 7 to 10. It can be seen from the figures that the high-solubility MCC solution prepared by the processes of the examples had no obvious sediment at the bottom of the bottle after standing, proving that the solution was stable and had good solubility; the MCC powder prepared by the processes of the comparative examples had poor solubility after reconstitution, obvious sediment could be observed at the bottom of the bottle, and only a small part could be dissolved in water.

[0092] 3. Centrifugal sedimentation rate test

[0093] With reference to the national standard test method GB 5413.29-2010, a method for quantifying the solubility of MCC powder was used to evaluate the solubility of the MCC powder. A lower centrifugal sedimentation rate indicates that more solute is dissolved in the solvent, and the powder has better solubility. The results are shown in Table 1.

[0094] Table 1

Sample Groups	Centrifugal Sedimentation Rate
Example 1	22.28%
Example 2	23.44%
Example 3	22.56%
Example 4	25.13%
Example 5	25.45%
Example 6	27.84%
Example 7	25.39%
Comparative Example 1	55.85%
Comparative Example 2	66.84%
Comparative Example 3	62.33%
Comparative Example 4	11.71%
Comparative Example 5	30.25%
Comparative Example 6	22.44%

[0095] 4. Dissolution Rate Test (Dispersibility Determination)

[0096] The test was performed with reference to the International Dairy Federation standard test method IDF 87:1979 to quantify the dissolution rate of the MCC powders. Higher dispersibility value indicates a faster dissolution rate. The results are shown in Table 2.

[0097] Table 2

Sample Groups	Dispersibility
Example 1	52.40%
Example 2	51.85%
Example 3	52.81%
Example 4	46.39%
Example 5	48.62%
Example 6	50.15%
Example 7	50.83%

Comparative Example 1	19.17%
Comparative Example 2	11.25%
Comparative Example 3	14.22%
Comparative Example 4	8.4%
Comparative Example 5	42.15%
Comparative Example 6	44.01%

[0098] 5. Cost-Effectiveness

[0099] The continuous production time and continuous powder production time for each Example and Comparative Example were recorded. The results are shown in Table 3.

[0100] Table 3

Sample Groups	Continuous Production Time /h	Continuous Powder Production Time /h
Example 1	8	4
Example 2	8	4
Example 3	8	4
Example 4	6	3
Example 5	7	3
Example 6	7	3
Example 7	8	3
Comparative Example 1	8	4
Comparative Example 2	8	4
Comparative Example 3	8	4
Comparative Example 4	/	/
Comparative Example 5	8	3
Comparative Example 6	2	0.5

[0101] From the above results, it can be seen that the casein powder prepared by the method of the present invention exhibits excellent solubility after reconstitution. Furthermore, the preparation method of the present invention boasts high production efficiency, strong continuous processing capability, and commercial cost-effectiveness.

[0102] Finally, it should be noted that the above embodiments are merely intended to illustrate the technical solutions of the present invention, rather than to limit the scope thereof. Although the present invention has been described in detail with reference to the foregoing embodiments, those of ordinary skill in the art should understand that modifications may still be made to the technical solutions described in the foregoing embodiments, or equivalent substitutions may be made for some technical features thereof. Such modifications or substitutions do not cause the essence of the corresponding technical solutions to depart from the spirit and scope of the technical solutions of the embodiments of the present invention.

Claims

[Claim 1]. A method for preparing a casein powder, characterized in that it comprises sequentially subjecting skim milk to microfiltration, ultrafiltration, and drying steps,

wherein in the microfiltration step, a volume concentration factor is 1 to 5; and

in the ultrafiltration step, an ultrafiltration membrane having a molecular weight cut-off of 30 kDa to 100 kDa is used, the concentration temperature is 10°C to 20°C, the concentration pressure is 0.1 to 1 mPa, and a total solids content after concentration is 10% to 25%.

[Claim 2]. The method for preparing a casein powder according to claim 1, characterized in that in the microfiltration step, a ratio of diafiltration water volume to retentate volume is 2:1 to 6:1; and

in the ultrafiltration step, a ratio of diafiltration water volume to total feed volume is 0.5:1 to 3:1.

[Claim 3]. The method for preparing a casein powder according to claim 1 or 2, characterized in that in the microfiltration step, a ratio of diafiltration water volume to retentate volume is 4.5:1; and

in the ultrafiltration step, a ratio of diafiltration water volume to total feed volume is 1:1.

[Claim 4]. The method for preparing a casein powder according to any one of claims 1 to 3, characterized in that in the microfiltration step, a microfiltration membrane with a pore size of 0.1 to 0.2 micrometers is used, the separation temperature is 40°C to 60°C, and the transmembrane pressure is 0.1 to 0.2 mPa.

[Claim 5]. The method for preparing a casein powder according to any one of claims 1 to 4, characterized in that the drying step is performed by using electrostatic spray drying.

[Claim 6]. The method for preparing a casein powder according to any one of claims 1 to 5, characterized in that in the electrostatic spray drying step, the atomizing nozzle uses a pressure atomizer; an atomization pressure is 0.1 to 2 mPa, an atomization temperature is 20°C to 60°C, a protective gas is nitrogen, an inlet air temperature is 60°C to 90°C, an outlet air temperature is 30°C to 50°C, an electrostatic field voltage is 1 to 20 kV, and a material preheating temperature is 30°C to 60°C.

[Claim 7]. The method for preparing a casein powder according to any one of claims 1 to 6, characterized in that the skim milk is obtained by standardizing raw bovine milk, followed by skimming and pasteurization.

[Claim 8]. The method for preparing a casein powder according to any one of claims 1 to 7, characterized in that a fat content in the skim milk is 0.01% to 0.1%.

[Claim 9]. A casein powder, characterized in that it is prepared by the method according to any one of claims 1 to 8.

[Claim 10]. The casein powder according to claim 9, characterized in that the casein powder has a centrifugal sedimentation rate of less than 28%, as determined according to GB 5413.29-2010; and

a dispersibility of greater than 46%, as determined according to IDF 87:1979.