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(54) Titre : PREPARATIONS SECHES CONTENANT DE L'HUILE PRODUITE PAR VOIE MICROBIENNE
(54) Title: DRY PREPARATIONS CONTAINING MICROBIALLY PRODUCED OIL

(57) Abrégé/Abstract:
The present invention is concerned with novel, stable cold water-dispersible pulverous preparations of a microbially produced oil, which is rich in arachidonic acid, said preparations containing fish gelatine as the matrix component and tocopherol, an ascorbic acid ester or a mixture of an ascorbic acid ester and tocopherol as the antioxidant.

Abstract

The present invention is concerned with novel, stable cold water-dispersible pulverous preparations of a microbially produced oil, which is rich in arachidonic acid, said preparations containing fish gelatine as the matrix component and tocopherol, an ascorbic acid ester or a mixture of an ascorbic acid ester and tocopherol as the antioxidant.

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The present invention is concerned with novel, stable, cold water-dispersible pulverous preparations of a microbially produced oil, referred to hereinafter as SCO (**S**ingle **C**ell **O**il), which is rich in **A**rachidonic **A**cid, referred to hereinafter as AA, as well as a process for their manufacture.

Cold water-dispersible preparations of fat-soluble substances play an important rôle in the field of human nutrition. Such preparations are usually commercialized in the form of emulsions or dry powders because of the water-insolubility of the fat-soluble active ingredients and the fact that they have a more or less pronounced instability and are difficult to handle. It is common in such preparations that the active ingredients, i.e. the fat-soluble substances, are generally enveloped by means of a matrix component. This matrix component is responsible, inter alia, for the protection of the active ingredient or for its stabilization, for an optimal resorption and for the water-dispersibility of the final preparation which may be required.

In accordance with the invention it has now been found that a preparation of the SCO referred to earlier, which is especially stable and which can be handled well, can be manufactured when fish gelatine is used as the protective colloid and an antioxidant is added. Antioxidants which are used are:

a) for the oil phase: tocopherol, an ascorbic acid ester or a mixture of an ascorbic acid ester and tocopherol, with the ascorbic acid ester being preferably ascorbyl palmitate;

b) for the aqueous phase: alkali or alkaline earth salts of ascorbic acid, preferably Na ascorbate.

The gelatine available under the name "Norland HiPure Fish Gelatin" from the firm Norland Products Inc., 695 Joyce Kilmer Ave., New Brunswick, N.J., USA, is an especially preferred fish gelatine.

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Any tocopherol can be used in the present invention, with examples of such tocopherols being α-tocopherol, γ-tocopherol or a mixture of natural tocopherols. In a preferred embodiment a mixture of natural tocopherols is used.

The ascorbyl palmitate and tocopherol are preferably used in a ratio of 1:5 to 1:25, especially 1:5, and the total amount which is added to the SCO is advantageously 1200-5250 parts per million, especially 1200-2000, preferably about 1200 parts per million. The ascorbyl palmitate and tocopherol can be added as a mixture or individually. When tocopherol is used alone, then the total amount is advantageously 1000-5000 parts per million, especially 1000-2000, preferably about 1000, parts per million.

The preparations in accordance with the invention can be manufactured in principle by preparing an aqueous emulsion of the SCO and the fish gelatine and converting this emulsion into a dry powder. This manufacturing process represents a further object of the present invention.

As a rule, the fish gelatine, and optionally adjuvants are firstly dissolved in water, which is conveniently accelerated by vigorous stirring. If desired, an antioxidant, for example Na ascorbate, can be added to the aqueous phase.

Usual adjuvants are, for example, mono- and di-saccharides; sugar alcohols; starch derivatives, e.g. maltodextrin; milk proteins, e.g. sodium caseinate; as well as plant proteins, e.g. soya protein, potato protein and wheat protein. Moreover, it has been found to be advantageous to carry out the dissolution of the fish gelatine at room temperature or elevated temperature, especially in the temperature range of 20°C to 90°C, preferably 50°C to 70°C. The so-called matrix is obtained in this manner. Then, the SCO stabilized by an antioxidant is emulsified in this matrix, advantageously by homogenization at atmospheric pressure or elevated pressure up to 1000 bar (100 MPa), preferably at 300-500 bar (30-50 MPa), or also using ultrasonics or similar technology. The pressure and the temperature are not critical parameters in this procedure, which can be carried out readily at temperatures of about room temperature to about 70°C, especially between about 60°C and 70°C, and atmospheric pressure.

The weight ratio of SCO to the other components (fish gelatine, sugar etc.) present in the final product is usually about 20:80 to about 80:20, with the precise ratio depending on the respective biological requirement of AA and on the need for a homogeneous and sufficiently fine distribution of the final preparations in the forms of application which are proposed for consumption.

The conversion of a thus-produced emulsion, which generally contains about 20 to about 80 weight percent of SCO depending on ingredients, into a dry powder can be effected e.g. by spray drying, the double dispersion process or also the starch catch process. In the latter process the sprayed emulsion droplets are caught in a starch bed and subsequently dried. Where required, the emulsion to be sprayed can be diluted with water.

In general, the preparations in accordance with the invention have a good cold water-dispersibility as well as a good flowability.

The preparations in accordance with the invention can be used for human nutrition, especially that of neonates.

The present invention is illustrated by the following Examples.

Example 1

48.5 g of dried fish gelatine and 48.5 g of crystalline sugar were placed in a 600 ml glass beaker. Then, 80 ml of deionized water were added and the mixture was brought into solution at 50°C while stirring with a mincer disc (1000 r/min.). Thereupon, 60 g of a SCO containing 50% AA, which had been stabilized with a mixture of 1000 ppm of a mixture of natural tocopherols and 200 ppm of acorbyl palmitate, was emulsified into this matrix and stirred for 15 minutes (during the emulsification and subsequent stirring the speed of the mincer disc was 4800 r/min.). After this time the internal phase of the emulsion has an average particle size of about 200 nm. The emulsion was then diluted with 90 ml of deionized water and heated to 65°C. 1 kg of starch fluidized with silicic acid was then placed in a laboratory spray tank and cooled to about 5°C. The emulsion was then sprayed into this using a rotating spray nozzle. The thus-obtained particles enveloped with starch were then sieved off from the excess starch and dried at room temperature

using compressed air. There were obtained about 190 g of dry powder with an AA content of 17.4%.

Example 2

44.2 g of dried fish gelatine, 44.2 g of crystalline sugar and 8.6 g of sodium ascorbate were placed in a 600 ml glass beaker. Further processing was carried out analogously to Example 1.

The average particle size of the internal phase was 180 nm. About 190 g of dry powder with an AA content of 17% were obtained.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. Stable, cold water-dispersible pulverous preparations of a microbially produced arachidonic acid containing oil, said preparations containing fish gelatine as the matrix component and tocopherol, an ascorbic acid ester or a mixture of an ascorbic acid ester and tocopherol as an antioxidant.

2. A preparation according to claim 1, which contains tocopherol or a mixture of ascorbyl palmitate and tocopherol as the antioxidant.

3. A preparation according to claim 1 or 2, which contains ascorbyl palmitate and tocopherol in a ratio of 1:5 to 1:25.

4. A preparation according to any one of claims 1-3, wherein the total amount of ascorbyl palmitate and tocopherol is 1200-5250 parts per million.

5. A preparation according to claim 4, wherein the total amount of ascorbyl palmitate and tocopherol is 1200-2000 parts per million.

6. A preparation according to any one of claims 1 to 5, wherein the tocopherol is a mixture of natural tocopherols.

7. A preparation according to any one of claims 1 to 6, which additionally contains sodium ascorbate and/or an adjuvant.

8. A process for the manufacture of stable, cold water-dispersible pulverous preparations of a microbially produced arachidonic acid containing oil, according to claim 1, which process comprises:

a) dissolving fish gelatine in water,

b) emulsifying the microbially produced oil stabilised by tocopherol, an ascorbic acid ester or a mixture of an ascorbic acid ester and tocopherol in the aqueous matrix and

c) converting the thus-produced emulsion into a dry powder.

9. A process as in claim 8 wherein in step (a) the water additionally contains sodium ascorbate and/or an adjuvant.

10. A process according to claim 8 or claim 9, wherein the emulsion produced in step b) is converted into a dry powder by normal spray drying, the double dispersion process or the starch catch process.