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# PREPARATION FOR HUMAN OR ANIMAL NUTRITION


## ABSTRACT

Novel, stable, cold water-dispersible, liquid or pulverous preparations of fat-soluble substances which contain fish gelatine as the protective colloid are described.

# PREPARATION FOR HUMAN OR ANIMAL NUTRITION

The present invention is concerned with novel, stable, cold water-dispersible preparations of fat-soluble substances and with a process for their manufacture.

The term "cold water-dispersible preparatons" means in the scope of the present invention liquid, as well as corresponding solid application forms. The solid application forms, i.e. preparations in pulverous form are preferred. The term "fat-soluble substances" embraces in the scope of the present invention especially the fat-soluble vitamins A, D, E and K, carotinoids such as e.g. beta-carotene, astaxanthin, apocarotenal, canthaxanthin, zeaxanthin etc. as well as poly-unsaturated fatty acids and the like. However, there will be immediately recognizable other fat-soluble substances which play a role in human or animal nutrition and, as the previously named, are usually marketed in the form of dry powders because of their water-insolubility or also their more or less pronounced stability and manipulability. Here there can be mentioned, in particular, oils and fats such as e.g. sunflower oil, palm oil, beef fat and the like. It is usually a common feature of such preparations that the active ingredients are enveloped with a protective colloid which is responsible, inter alia, for the protection of the active substance or for its stabilization, for an optimal resorption and for the water-dispersibility of the final preparation which may be required. As the protective colloid there is normally used gelatine which originates from warm-blooded animals and which accordingly also has certain disadvantages. Merely by way of example there are to be mentioned here the fact that,

for example, preparations based on such gelatine can not be used worldwide for religious reasons, that, without an expensive manufacturing process, this gelatine, and accordingly also the pulverous preparations manufactured therewith, does not always have the desired dispersibility in cold water, etc.

In accordance with the invention it has now been established that all of these disadvantages can be eliminated when fish gelatine is used in place of gelatine from warm-blooded animals.

The stable, cold water-dispersible preparations of fat--soluble substances in accordance with the invention accordingly contain fish gelatine as the protective colloid.

These preparations can be manufactured in a manner known per se such as, for example, by preparing an aqueous emulsion of the active substance and a protective colloid and if desired subsequently converting this into a dry powder. In the process in accordance with the invention fish gelatine is, however, used as the protective colloid.

The fish gelatine which is used in the scope of the present invention can be prepared in principle in a manner analogous to the gelatine of warm-blooded animals, but here fish skin is used exclusively. Moreover, skin of deep-sea fishes such as, for example, cod, shellfish, torsk etc is preferred. Such a fish gelatine has a gelling point below about 20°C, and particularly between about 5°C and about 10°C; this is in contrast to gelatine from warm-blooded animals which gels at about 35°C. An especially preferred fish gelatine is the gelatine obtainable under the name "Norland HiPure Liquid Gelatin" from the firm Norland Products Inc., 695 Joyce Kilmer Ave., New Brunswick, N.J., USA.

As mentioned previously, the preparations in accordance with the invention can be manufactured in a manner known per se. This is normally effected by emulsifying the active substance or the active substances in a matrix with a subsequent drying of the so obtained emulsion.

In the manufacture of the emulsion there can, of course, be used in addition to fish gelatine, which serves not only as an emulsifier but also as a protective colloid, additional adjuvants which are normally used in such preparations. As examples of these there can be named sugars such as, for example, saccharose, sugar alcohols, starch derivatives such as maltodextrin, milk proteins such as, for example, sodium caseinate or also vegetable proteins such as e.g. soya protein, potato protein, wheat protein etc.

As a rule, all ingredients, except the active substance, are firstly dissolved in water, whereby the so-called matrix is obtained. Then, the active substance or the active substances is/are emulsified in this matrix. The preparation of the emulsion can be effected in a manner known per se, for example by vigorous stirring or also by means of ultrasonics and the like. The pressure and the temperature are not critical parameters in this procedure and the entire operation can be carried out readily at temperatures from about room temperature up to about 70°C and under atmospheric pressure.

The ratio of oil phases (fat-soluble substances) to the accompanying substances ultimately present in the end product generally amounts from about 1%:99% to about 60% to about 40%. The precise ratios depend on the actual biological requirement with respect to active substances and on the demand for uniform and sufficiently fine distribution of the final preparations in the forms of use which are proposed for consumption. In the event that stabilizing substances are also required or desired in the preparations,

then these can generally be dissolved in the oil phase. As already mentioned, the fish gelatine also serves as an emulsifier in the preparation of the emulsion. However, further emulsifiers can also be used, whereby here there comes into consideration primarily e.g. ascorbyl palmitate which then, moreover, also serves as a stabilizer.

The conversion of a thus-prepared emulsion into a dry powder can also be effected in a manner known per se, e.g. by normal spray-drying, by a double-dispersion process or also by a starch-catch process. In the latter process the sprayed emulsion droplets are collected in a bed of starch and subsequently dried.

The preparations in accordance with the invention can be used not only in animal nutrition but also for human nutrition. In certain instances it can also be convenient not to convert the emulsions prepared by means of fish gelatine firstly into a dry powder, but to use them directly as such.

The term "fish gelatine" in the following Examples signifies in each case the "Norland HiPure Gelatin" originating from the firm Norland.

## Example 1

144 g of fish gelatine (as an about 45% aqueous solution) and 97.2 g of crystalline sugar are placed in a 600 ml glass beaker. Then, 20 ml of distilled water are added and the mixture is brought into solution while stirring with a blade stirrer (2800 r/min.) at 40°C. Thereupon, 100 g of vitamin A palmitate (1.7 million IU/g and stabilized with α-tocopherol) are emulsified in this matrix and stirred for a further 60 minutes. After this time the internal phase of the emulsion has an average particle size of about 0.6 µ. The emulsion is then diluted with 100 ml of

distilled water and heated to 65°C. Then, about 1 kg of starch, fluidized by means of silicic acid, are placed in a laboratory spraying tank and cooled to about 5°C. The emulsion is now sprayed into this using a rotary spray nozzle. The thus-obtained particles, which are enveloped with starch, are then sieved off from the excess starch and dried at room temperature using compressed air. There are obtained about 330 g of dry powder having a vitamin A content of 530,000 IU/g.

## Example 2

An emulsion is prepared in an analogous manner to Example 1 starting from 117 g of fish gelatine (as an about 45% aqueous solution), 58.4 g of crystalline sugar, 20 ml of distilled water and 13.5 g of vitamin A palmitate (1.7 million IU/g, stabilized with α-tocopherol). This emulsion is diluted with 70 ml of water. The average particle diameter of the internal phase amounts to about 0.3 µ. After drying there are obtained 160 g of dry powder having a vitamin A content of 139,600 IU/g.

## Example 3

An emulsion is prepared in an analogous manner to Example 1 starting from 51.2 g of fish gelatine (as the dry substance), 76.8 g of maltodextrin MDO5 (from the firm Roquettes Freres, Lille, France), 80 ml of distilled water, 31.9 g of an oily solution of 25 g of vitamin A acetate (2.8 million IU/g) and 2.5 g of α-tocopherol in 4.4 g of arachis oil. This emulsion is diluted with 90 ml of distilled water. The average particle diameter of the internal phase amounts to 0.28 µ. After the drying operation there are obtained 195 g of dry powder having a vitamin A content of 351, 300 IU/g.

## Example 4

An emulsion is prepared in an analogous manner to Example 1 starting from 31.3 g of fish gelatine (as an about 45% aqueous solution), 42.3 g of maltodextrin MDO5 (from the firm Roquettes Freres, Lille, France), 20 ml of distilled water and 63.6 g of tocopherol acetate. This emulsion is diluted with 200 ml of water. The average particle diameter of the internal phase amounts to 0.34 µ. This emulsion is then spray-dried in a laboratory spray dryer from the firm Büchi, Flawil, Switzerland. The inlet temperature amounts to 186°C and the outlet temperature to 106°C. There are thus obtained 115 g of dry powder having a tocopherol acetate content of 52.1%.

## Example 5

An emulsion is prepared in an analogous manner to Example 1 starting from 28.5 g of fish gelatine (as the dry substance), 42.7 g of maltodextrin MDO5 (from the firm Roquettes Freres, Lille, France), 50 ml of distilled water and 84.8 g of an oily solution of 84 g of gamma-linolenic acid (as the triglyceride) and 0.8 g of α-tocopherol. This emulsion is diluted with 85 ml of water. The average particle diameter of the internal phase amounts to 0.4 µ. After the drying operation there are obtained 200 g of dry powder having a content of gamma-linolenic acid of 9.8%.

## Example 6

a) 18 g of fish gelatine (as the dry substance), 27 g of maltodextrin MDO5 (from the firm Roquettes Freres, Lille, France) and 14.7 g of crystalline sugar are dissolved in 180 ml of distilled water at 70°C in a 1 L glass beaker. Then, 5 g of ascorbyl palmitate are added to the solution while stirring and the pH of the solution is adjusted to 7.5 ± 0.2 by means of 20%

sodium hydroxide solution.

b) 13 g of ß-carotene, 5.5 g of arachis oil and 1.5 g of α-tocopherol are dissolved in 200 ml of chloroform in a 500 ml round flask during 15 minutes on a steam bath.

c) The ß-carotene solution obtained in accordance with b) is emulsified in the solution prepared in accordance with a) in a 2 L round flask for 30 minutes at 40°C. After this time the internal phase has a particle size of about 0.18 µ. The chloroform is now removed in a short-path distillation apparatus at 50°C under a water-jet vacuum and the emulsion is sprayed into starch in an analogous manner to Example 1. There are obtained 85 g of dry powder having a ß-carotene content of 12.5%.

## Example 7

An emulsion is prepared in an analogous manner to Example 1 starting from 56.4 g of fish gelatine (as the dry substance), 84.6 g of maltodextrin MD05 (from the firm Roquettes Freres, Lille, France), 125 ml of distilled water and 159 g of sunflower oil. This emulsion is diluted with 242 ml of distilled water. The average particle diameter of the internal phase amounts to about 0.3 µ. This emulsion is then spray-dried in a transportable minor laboratory spray dryer from the firm NIRO Atomizer, Söborg, Denmark. The inlet temperature amounts to 200°C and the outlet temperature to 90-94°C. There are thus obtained 230 g of dry powder having an oil content of 53%.

## Example 8

An emulsion is prepared in an analogous manner to Example 1 starting from 56.4 g of fish gelatine (as the dry substance), 84.6 g of maltodextrin MS05 (from the firm Roquettes Freres, Lille, France), 125 ml of distilled water

and 159 g of beef fat (stabilized with 100-200 ppm of tocopherol). This emulsion is diluted with 242 ml of distilled water. The average particle diameter of the internal phase amounts to about 0.5 µ. This emulsion is then spray-dried in a transportable minor laboratory spray drier from the firm NIRO Atomizer, Söborg, Denmark. The inlet temperature amounts to 200°C and the outlet temperature to 90-94°C. There are thus obtained 235 g of dry powder having a fat content of 53%.

Example 9

An emulsion is prepared in an analogous manner to Example 1 starting from 56.4 g of fish gelatine (as the dry substance), 84.6 g of maltodextrin MD05 (from the firm Roquettes Freres, Lille, France), 125 ml of distilled water and 159 g of palm oil. This emulsion is diluted with 242 ml of distilled water. The average particle diameter of the internal phase amounts to about 0.3 µm. This emulsion is then spray-dried in a transportable minor laboratory spray drier from the firm NIRO Atomizer, Söborg, Denmark. The inlet temperature amounts to 200°C and the outlet temperature to 90-95°C. There are thus obtained 225 g of dry powder having an oil content of 53%.

CLAIMS:

1. In a stable, cold water dispersible, liquid or pulverous preparation of fat-soluble substances for animal or human nutrition which fat-soluble substances are stabilized or enveloped witha gelatine as a protective colloid, the improvement comprising using as the gelatine protective colloid a fish gelatine.

2. Preparations in accordance with claim 1, which are pulverous and wherein the fat-soluble substance(s) is/are enveloped with fish gelatine as the protective colloid.

3. Preparations in accordance with claim 1 which contain vitamin A, D, E or K or a carotinoid or a polyunsaturated fatty acid as the fat-soluble substance.

4. Preparations in accordance with claim 1 which contain oils or fats as the fat-soluble substance.

5. Preparations in accordance with claim 4, wherein the oil or fat used as the fat-soluble substance is sunflower oil, palm oil or beef fat.

6. A process for the manufacture of stable, cold water-dispersible, liquid or pulverous preparations of fat-soluble substances comprising the steps of dissolving fish gelatine and all the other ingredients except the active substance(s) [fat-soluble substance(s)] in water to obtain a matrix, and admixing the active substance(s) with said matrix by vigorous stirring or ultrasonics at a temperature from about room temperature up to about 70°C and under atmospheric pressure to achieve emulsification of the active substance(s) in the matrix, thereby forming an aqueous emulsion of the active substance(s) and a protective colloid of fish gelatine.

7. A process in accordance with claim 6 for the manufacture of pulverous stable, cold water-dispersible preparations of fat-soluble substances, comprising the subsequent step of drying the so-manufactured emulsion by spray-drying or applying a double-dispersion or starch-catch process and thus converting the emulsion into the desired dry powder form of the preparation.

8. A process in accordance with claim 6, wherein vitamin A, D, E or K, a carotinoid or a polyunsaturated fatty acid is used as the fat-soluble substance.

9. A process in accordance with claim 6, wherein an oil or fat is used as the fat-soluble substance.

10. A process in accordance with claim 9, wherein the oil or fat used as the fat-soluble substance is sunflower oil, palm oil or beef fat.

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