SOLID PRODUCT CONTAINING PROPOLIS COMPONENTS, AND ITS PREPARATION AND USES

The present invention relates to a solid product containing propolis components which has a satisfiable water-dispersibility, and its preparation and uses, more particularly, it relates to a process for preparing a solid product containing propolis components which are soluble in a readily water-soluble organic-solvent, said process comprising incorporating propolis components in one or more saccharides selected from anhydrous saccharides and cyclodextrins; and it relates to a process for preparing a solid product containing propolis components which are soluble in a readily water-soluble organic-solvent, said process containing a step of incorporating an aqueous solution containing propolis components, which are soluble in a readily water-soluble organic-solvent, in one or more saccharides selected from anhydrous saccharides and cyclodextrins to effect dehydration and solidification.

As described in Propolis in Natural Therapeutics, published by Librairie Maloine S.A. Editeur, Paris, France (1983) and Fragrance Journal, No.83, pp.20-28 and pp.36-43

(1987), propolis, which contains resins, balms, beeswaxes, essential oils, pollens and flavonoids, is a resin-like product stored in beehives by bees, and has been used from old times in folk medicines such as prophylactic- and therapeutic-agents for diseases of circulatory-, respiratory-, digestive-, genital-, dermatopathic-, mental- and nervous-systems.

Propolis is a product in the form of mass or lump and the main components thereof are hydrophobic or scarcely soluble in water, and these render its intact use difficult, and, usually propolis has been used as a propolis extract in liquid (or an alcoholic tincture of propolis) which is prepared by extracting intact propolis with a relatively high-concentration of a readily water-soluble organic-solvent such as ethanol.

As described in Japanese Patent Laid-Open No.245,159/90, the following drawbacks are, however, inevitable when such propolis extract is used as a food product for health:

(1) When a propolis extract is diluted with water, propolis components, which are soluble in a readily water-soluble organic-solvent, are ununiformly precipitated, coagulated or solidified into a lump or mass;

(2) When a propolis extract is orally taken, a relatively-high concentration of a readily water-soluble organic-solvent contained in the extract, as well as the components dissolved in

the solvent, strongly stimulate oral mucosa, and after the intake the propolis extract is first diluted with gastric, then the components are similarly as with water ununiformly precipitated to cause an unfavorable feeling such as stickiness in the mouth;

(3) A person who is allergic to an organic solvent could not use a propolis extract as a food product for health; and

(4) A propolis extract is in the form of liquid, and this hinders its handleability and portability.

In order to improve the above drawbacks, several proposals are made: For example, Japanese Patent Laid-Open No.197,523/86 proposes a process, i.e. "A process for preparing a product containing propolis which has a satisfiable water-dispersibility, c h a r a c t e r i z e d in that it comprises dewaxing a liquid propolis-extract which has been prepared by extracting intact propolis with a readily water-soluble organic-solvent, crystallizing and solidifying components in the extract having an antibacterial activity by using a water-soluble filler, adding to the resultant an emulsifier and an antioxidant, and drying the resultant mixture."; and Japanese Patent Laid-Open No.245,159/90 proposes a composition, i.e. "A food composition of propolis, characterized in that it comprises propolis components which are soluble in a monohydric alcohol, a medium containing

OH-base which can form multiple hydrogen-bonds, and a surfactant in the form of polyol and fatty acid ester, wherein said surfactant is contained in said food composition in the range of 0.01-25 parts by weight when the total amount of said propolis components and said medium is 100 parts by weight."

It was elucidated that these proposals, however, improved the water dispersibility of propolis components, but had the drawbacks of that the processes were complicated and their final products had an unsatisfiable taste inherent to the emulsifier and surfactant which were inevitably used in the processes.

It has been a strong demand to overcome the drawbacks and to establish a solid product containing propolis components which can be prepared without using any emulsifier or surfactant, said solid product having a satisfiable water-dispersibility, taste preference, handleability and portability.

The present invention aims to overcome the above drawbacks, more particularly, the present inventors studied a process without using any emulsifier or surfactant to convert propolis components as the main components of propolis, which are hydrophobic or scarcely soluble in water but soluble in a readily water-soluble organic-solvent, into a solid product

having a satisfiable water-dispersibility and taste preference.

As a result, the present inventors found that a solid product containing propolis components, prepared by incorporating propolis components which were soluble in a readily water-soluble organic-solvent in one or more saccharides selected from anhydrous saccharides and cyclodextrins, had a satisfiable water-dispersibility and taste preference, and could completely overcome conventional drawbacks. Thus, the present inventors accomplished the present invention.

Accordingly, the present invention provides a readily water-dispersible solid product which comprises a saccharide(s) and propolis components, which are contained in intact propolis or dewaxed propolis preparations and soluble in an aqueous solution of an organic solvent. The solid product is prepared by a process comprising:

(a) selecting a quantity of material selected from the group consisting of intact propolis and dewaxed propolis preparations;

(b) extracting said quantity of material with an aqueous solution of an organic solvent selected from the group consisting of acetone, acetic acid and alcohols, wherein the concentration of said organic solvent in said aqueous solution is 30 w/w % or higher to form a first extract containing propolis components which are contained in said quantity of material and soluble in said organic solvent;

(c) evaporating water from the first extract containing said propolis components to give a moisture content of 1 w/w % or higher but lower than 50 w/w % to form a second extract;

(d) incorporating the second extract, which contains said propolis components, in one or more saccharides selected from the group consisting of anhydrous saccharides and cyclodextrins in order to dehydrate the second extract, wherein the amount of said extract against that of said saccharide(s) is in the range of 0.1-50 w/w %, on a dry solid basis;

(e) drying the dehydrated second extract in the step (d) at ambient temperature; and

(f) incorporating the resultant extract in the step (e) in an effective amount of one or more saccharides selected from the group consisting of anhydrous saccharides and cyclodextrins to obtain a water-dispersible solid product.

A solid product obtainable by the process is also provided.

The anhydrous saccharides usable in the invention are those which can be converted into a hydrous crystal in the presence of water while exerting a dehydrating activity: for example, anhydrous maltose, lactitol, glucose, galactose, paratinose, raffinose, erlose and melezitose as disclosed in Japanese Patent Laid-Open Nos. 136,240/88, 152,535/88, 152,536/88 and 152,537/88 can be used in the invention.

These saccharides are advantageously used in the form of anhydrous

crystalline saccharide with the possible-lowest level of moisture, preferably, a moisture content of 3 w/w % or lower, more preferably, a moisture content of 2 w/w % or lower, and the most satisfiable saccharide is "FINETOSE®", a crystalline α-maltose powder commercialized by Hayashibara Co., Ltd., Okayama, Japan, as disclosed in Japanese Patent Laid-Open No.35,800/86.

The cyclodextrins usable in the invention are α-, β- and γ-cyclodextrins, and one or more of them are suitably used in the invention.

If necessary, a partial starch hydrolysate containing a cyclodextrin together with saccharides such as maltooligosaccharides, dextrins and branched dextrins can be suitably used in the invention.

In the present preparation of a solid product, a cyclodextrin powder or a partial starch hydrolysate containing a cyclodextrin with the possible-lowest level of moisture, preferably, a moisture content of 3 w/w % or lower, more preferably, a moisture content of 2 w/w % or lower, can be advantageously used in the invention.

The wording "propolis components which are soluble in a readily water-soluble organic-solvent" as referred to in the invention are those which are contained in intact propolis and dissolvable in a readily water-soluble organic-solvent, and the preparations thereof are those which contain a step of extracting intact propolis or a dewaxed propolis-preparation,

which has been prepared by dewaxing intact propolis with a solvent such as ethyl ether and ethyl methyl ether, with a readily water-soluble organic-solvent, for example, acetone, acetic acid and alcohols such as methanol, ethanol and propanol, with a relatively-high concentration of aqueous solutions of the organic solvents, i.e. 30 w/w % or higher, at an ambient temperature or under heating conditions, or, if necessary under refluxing conditions.

If necessary, intact propolis can be boiled into a suspension containing the propolis components, or, the α-glycosyl flavonoids as described in the specification which was applied by the present inventors to the Japanese Patent Office on April 11, 1991, entitled "α-Glycosyl flavonoids, and their preparation and uses", can be used as the propolis components.

These propolis components in the form of solution or suspension usually contain a satisfiable level of moisture required in the present preparation of a solid product. When the moisture level of the propolis components is insufficient, an adequate amount of water can be advantageously added thereto.

In the present preparation of a solid product, the wording "incorporating in a saccharide a solution containing propolis components which are soluble in a readily water-soluble organic-solvent", means processes which can incorporate the solution in the saccharide to homogeneity by stirring or mixing: For a satisfiable example, a process

containing a step of incorporating an ethanol solution, which contains propolis components, in one or more saccharides selected from anhydrous saccharides and cyclodextrins can be favorably used, wherein the amount of said ethanol solution against that of the saccharide(s) is in the range of 0.1-50 w/w %, on the dry solid basis (d.s.b.), more preferably, in the range of 0.2-30 w/w %, d.s.b.

The administrable amount of propolis components is relatively too low when the amount of the ethanol solution containing propolis components is less than 0.1 w/w %, d.s.b., and the water dispersibility of a solid product is difficult when the amount of propolis components is 50 w/w %, d.s.b., or higher.

In case of incorporating in a saccharide an aqueous solution containing propolis components, coexistence of a relatively-low level of moisture in the aqueous solution, 1 w/w % or higher but lower than 50 w/w %, facilitates a homogeneous dispersion and incorporation of the propolis components in the saccharide, and enables a relatively-large amount of propolis components to be readily adsorbed to, dispersed in or included by the saccharide. Thus, the content of the propolis components in the resultant solid product is increased, and the water dispersibility is highly improved.

In order to dehydrate an aqueous solution containing propolis components which are soluble in a readily

water-soluble organic-solvent into a solid product by incorporating the aqueous solution in a saccharide, the coexisting moisture in the aqueous solution is dehydrated by one or more saccharides selected from anhydrous saccharides and cyclodextrins.

To effectively promote the dehydration step, the resultant mixture may be further admixed with one or more saccharides selected from anhydrous saccharides and cyclodextrins, or, if necessary the resultant mixture can be dried by heating.

Since the effective components of propolis are readily volatiled, deteriorated or decomposed by severe treatments such as an irradiation of ultraviolet ray and a heating at a relatively-high temperature, it is recommendable to employ a process wherein a dehydrating action of a saccharide at an ambient temperature is mainly utilized in order to stably retain the effective components as much as possible, or, if necessary a readily water-soluble organic-solvent can be removed by evaporation under the conditions of that they do not give any undesirable damage to the effective components.

For such proposes, one or more saccharides selected from anhydrous saccharides and cyclodextrins with the possible-lowest level of moisture are advantageously used.

The solid products thus obtained are usually in the from of powder.

If necessary, the solid products can be further subjected to pulverization- or spherization-step.

The solid product according to the present invention is a solid product containing propolis components which has a satisfiable water-dispersibility, taste preference and handleability.

It was found that the present solid product similarly as conventional propolis extracts exerted a satisfiable efficacy in proportion to the content of propolis components which were hydrophilic and soluble in an organic solvent. Thus, the present solid product can be advantageously used as a food product for health, antibacterial agent and therapeutic agent in the maintenance and improvement of health, and the prevention and treatment of diseases such as virus diseases, bacterial diseases, traumatic diseases, immunopathies, rheumatisms, diabetics, diseases of circulatory organs, malignant tumors and nervous diseases, as well as in the promotion of recovery of health from such diseases.

In use, the present solid product is orally administered at a dose of about 0.01-5g/day/adult, based on the weight of propolis components, in one or several shots, or, if necessary the solid product can be formed into ointment for parenteral use.

It was also found that the present solid product could be favorably used as an agent such as a flavor-imparting agent, deodorant and agent for urine therapy.

Urine therapy (uropathy) is described in Kiseki ga okoru Nyo-Ryoho, edited by Ryoich Nakao, published by Makino Publisher, Tokyo, Japan (1990).

According to the book, it has been known that the maintenance and improvement of health, and the prevention and treatment of diseases, as well as the promotion of recovery of health from diseases, can be effectively attained by daily drinking about 100-200ml of urine.

It has been also known that such effects will be expected in proportion to the intake of urine without causing no side effect.

Unpleasant smell and taste of urine, however, hinder a person to try urine therapy, or these make him or her to be strongly reluctant to directly drink urine.

It was found that the present solid product when dissolved in a fresh urine readily dispered or dissolved in it and imparted it a propolis flavor to diminish or lower the unpleasant smell of urine, as well as improving the taste of urine and augmenting the effect of urine therapy. Thus, the present solid product can be advantageously used as an agent for urine therapy.

The objectives are attained by using about 0.1-5g of the present solid product in powder against 100ml of a fresh urine.

The present solid product can be advantageously formed into a product such as a granule, short-rod and tablet

to meet to its final use, or can be processed into an ointment wherein the solid product is incorporated.

When the solid product is used as a sublingual agent or an external application, it can be formed, for example, into a tablet, ointment or cataplasm.

If necessary, the present solid product can be derived from animals and plants such as royal jelly, cod-liver oil, egg oil, oyster extract, turtle extract, "mamushi" (a pit viper) extract, ginseng extract, Saururaceae extract, Ginkgoaceae extract, pine-leaf extract, leaf extract of Sasa albo-marginata, extract of Japanese apricot, loquat-leaf extract, field-horsetail extract, Spirulina extract and chlorella extract; vitamins such as thiamine, riboflavin, vitamin B_{17}, L-ascorbic acid, α-glycosyl L-ascorbic acid, rutin, α-glycosyl rutin, carotenoid, ergosterol and tocopherol; hormones such as isulin, growth hormone, urogastrone, erythropoietin, calcitonin, prolactin and sex hormones (androgen and estrogen); and biologically active substances including cytokines such as interferon, lymphotoxin, tumor necrosis factor, macrophage migration inhibitory factor, colony stimulating factor, transfer factor and interleukin 2. If necessary, one or more of these additives can be advantageously used in the present solid product.

The following Experiments will explain a physiological activity of the present solid product containing propolis components.

[Experiment 1]

Preparation of solid product containing propolis components

To one part by weight of a dewaxed propolis extract having about 30 w/w % propolis components prepared in accordance with the method in Example 5 was added 2 parts by weight of an anhydrous crystalline maltose powder, and the resultant mixture was ventilated and dried at 40°C to obtain a solid product in powder containing propolis components.

By using the solid product thus obtained, the actions or activities of the present solid product were studied.

[Experiment 2]

Virus inhibitory effect

Virus inhibitory effect of the present solid product was studied with the plaque-reduction assay referred to as a plaque-depressing-dose-50% (PDD_{50}) wherein FL cells were infected with vesicular stomatitis virus (VSV) or herpes simplex virus type 1 (HSV-1) which had been treated with a solution containing propolis components.

A propolis solution containing 10 or 50μg/ml of propolis components was prepared by diluting with or dissolving in Hanks' solution (pH about 7.4) a solid product in powder containing propolis components prepared by the method in Experiment 1. As a virus solution, a solution containing virus with a concentration of about 10^2-10^4 plaque-forming-units (PFU) per ml was prepared. Half ml aliquots of the propolis solution and the virus solution were mixed, and FL cells were

infected to form plaques with viruses which had been treated with the mixture solution at 37°C for an hour, followed by counting the resultant plaques.

As a control, a virus solution free of propolis components was prepared, and FL cells were infected similarly as above with the virus solution to form plaques, followed by counting the resultant plaques.

Virus inhibitory rate (%) was calculated by the following formula:

$$\text{Virus inhibitory rate (\%)} = \frac{(\text{Control plaques}^{*}) - (\text{Plaques}^{*} \text{ of propolis treatment})}{\text{Control plaques}^{*}} \times 100$$

Note : In the formula, "plaques" indicates the number of plaques.

The relationship between the concentration of propolis components (μg/ml) and the virus inhibitory rate (%) was summarized in Table 1.

Table 1

Virus	Form	Propolis components Concentration (μg/ml)		
		0	10	50
VSV	Extract	0	29.4	48.3
	Solid	0	29.3	48.7
HSV-1	Extract	0	100	100
	Solid	0	100	100

As evident from the results in Table 1, it was elucidated that the coexistence of propolis components exerted a strong virus-inhibitory-effect, and the level of which increased as the increase of the concentration of the propolis components.

It was elucidated that the virus inhibitory effect of the present solid product depended on the amount of propolis components therein, and the effect was almost the same level as that of conventional propolis extracts.

[Experiment 3]

Stress inhibitory effect

[Experiment 3-1]

Load of Stress on rat

In accordance with the method described by Matsuo et al. in Shin-yaku Kaihatsu no tameno Dobutsu Model Riyo Shusei, edited by Ryuta Ito, Ryo Takahashi and Nishio Honda, pp.247-254 (1985), published by R&D Planing, Tokyo, Japan, male rats of Wister strain, 280-350g each, were restrained in a stress cage made of wire nets, and soaked the whole body except for the head in a 23°C water for 18 hours to induce an acute ulcer.

[Experiment 3-2]

Oral administration of stress-inhibitory-agent

Rats were administered with aqueous solutions as a stress-inhibitory agent, said aqueous solutions being prepared by dissolving in distiled water a solid product containing propolis components obtained by the method in Experiment 1 to give prescribed concentrations. As a control, other rats were administered with distiled water or an aqueous solution which had been prepared by dissolving an anhydrous crystalline maltose in distiled water to give a prescribed concentration.

The administration method used in this Experiment was that each rat in a group consisting of 8 rats was compulsorily administered orally with 3ml aliquots of stress inhibitory agents with a sound for stomach at 10 minutes before the initiation of loading a stress on rats.

The relationship among rat groups, stress inhibitory agents and doses was summarized in Table 2.

Table 2

Group	Stress inhibitory agent	Dose(mg)/3ml/rat	Rat (Head)
I	Distiled water		8
II	Anhydrous crystalline maltose	9.0	8
III	Solid product containing propolis components	0.3	8
IV	Solid product containing propolis components	0.9	8
V	Solid product containing propolis components	3.0	8
VI	Solid product containing propolis components	9.0	8

[Experiment 3-3]

Evaluation of effect

After completion of loading a stress on rats, which were then allowed to inhale ether to die and anatomized to measure the length (mm) of each erosion formed on the surface of stomach mucosa, followed by summing up the lengths to

give the ulcer index of each rat.

The results were summarized in Table 3.

Table 3

Group	Stress inhibitory agent	Dose(mg)/3ml/rat	Ulcer-index (mm)
I	Distiled water		63
II	Anhydrous crystalline maltose	9.0	56
III	Solid product containing propolis components	0.3	38
IV	Solid product containing propolis components	0.9	38
V	Solid product containing propolis components	3.0	36
VI	Solid product containing propolis components	9.0	27

Note : "Ulcer index" is a mean value of a group of 8 rats.

As evident from Table 3, the ulcer indexes of the rats in the groups which had been administered with a solid product containing propolis components were as follows: The ulcer indexes in the groups III to V, which had been administered with the solid product at a dose in the range of 0.3-3.0mg per rat, were lowered to about 60% against that of

the group I as a control with distiled water, and the ulcer index in the group VI with a dose of 9.0mg/rat of the solid product was lowered to about 40% against that of the group I. These elucidated that the present solid product exerted a strong stress-inhibitory-effect. Statistically speaking, the group II which had been administered with anhydrous crystalline maltose showed no significant difference against the group I with distiled water, while the occurrence of an acute gastric ulcer in the groups III and V was significantly lowered ($p<0.05$), and the occurrence in the group IV was significantly lowered ($p<0.01$). Thus, the present solid product evidently exerts a stress inhibitory effect.

The following Examples are the preferred embodiments of the present invention, and it is to be understood that the present invention is not limited by such Examples.

[Example 1]

Intact propolis was extracted in an usual manner with a relatively-high concentration of an aqueous ethanol solution to obtain a propolis extract having about 65 w/w % ethanol, about 20 w/w % moisture and about 15 w/w % propolis components, and 5 parts by weight of the propolis extract thus obtained was admixed with 2 parts by weight of γ-cyclodextrin and 5 parts by weight of an anhydrous crystalline maltose powder. The resultant mixture was first ventilated and dried at 40°C for an hour, then mixed to homogeneity with 7 parts by weight of an anhydrous crystalline maltose into a solid product in powder.

One g aliquots of the solid product were injected into laminated aluminum-bags and sealed.

The product having a satisfiable water-dispersibility and taste preference can be advantageously used as a food product for health, antibacterial agent, therapeutic agent, flavor-imparting agent, deodorant and agent for urine therapy in the maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

When the product is used as a food product for health, it can be taken alone or used by dispersing or dissolving about 0.2-1g thereof in 200ml of tea, milk or juice, prior to use.

When the product is used as an agent for urine therapy, it can be used by dispersing or dissolving about 0.5-2g thereof in 100ml of a fresh urine, prior to use.

[Example 2]

Intact propolis was extracted in an usual manner with a relatively-high concentration of an aqueous ethanol solution to obtain a propolis extract having about 74 w/w % ethanol, about 8 w/w % moisture and about 18 w/w % propolis components, and 5 parts by weight of the propolis extract thus obtained was admixed with an adequate amount of lemon flavor and 5 parts by weight of "DEXY PEARL® SD-20", a partial starch hydrolysate containing α-, β- and γ-cyclodextrins, commercialized by Ensuiko Seito Kabushiki Kaisha, Yokohama, Japan. The mixture

was first ventilated and dried at 40°C for an hour, then admixed to homogeneity with 0.02 parts by weight of "α-G Sweet", a sweetener of stevioside, commercialized by Toyo Sugar Refining Co., Ltd., Tokyo, Japan, 0.02 parts by weight of citric acid, and 15 parts by weight of an anhydrous crystalline maltose powder, and the resultant mixture was subjected to a granulator to obtain a solid product in granule. One hundred g aliquots of the solid product were injected into containers and sealed.

Similarly as the product in Example 1, the product having a satisfiable water-dispersibility and taste preference can be advantageously used as a food product for health, antibacterial agent and agent for urine therapy in the maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

[Example 3]

A solid product in granule was obtained similarly as in Example 2 except for that the anhydrous crystalline maltose powder in Example 2 was replaced with an anhydrous crystalline lactitol powder, and the product was injected in bottles and sealed.

Similarly as the product in Example 1, the product having a satisfiable water-dispersibility and taste preference can be advantageously used as a food product for health, antibacterial agent and agent for urine therapy in the

maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

[Example 4]

A solid product in granule prepared by the method in Example 2 was subjected in an usual manner to a tabletting machine to obtain a tablet, about 800mg each.

The product is suitably used as a food product for health, especially, as a sublingual tablet because the product gradually releases propolis components in the mouth, followed by the absorption of the propolis components via mucosa.

The product is favorably used as a cachou because you can continuously luxuriate in a propolis flavor.

[Example 5]

Intact propolis was extracted in an usual manner with a relatively-high concentration of an aqueous methanol solution to obtain a propolis extract, which was then evaporated, dried up, dewaxed by the solid-liquid separation method using ethyl ether, and dissolved in a relatively-high concentration of an aqueous ethanol solution to obtain a dewaxed propolis extract having about 70 w/w % ethanol, about 10 w/w % moisture and about 20 w/w % propolis components. Five parts by weight of the dewaxed propolis extract thus obtained was admixed with an adequate amount of a herb flavor and 5 parts by weight of a partial starch hydrolysate containing α-, β- and Ь-cyclodextrins, and the mixture was ventilated and dried at

30°C for 2 hours, and further admixed to homogeneity with 0.5 parts by weight of calcium L-ascorbate and 20 parts by weight of an anhydrous crystalline maltose powder to obtain a solid product in powder. Two g aliquots of the solid product were injected in containers and sealed.

Similarly as the product in Example 1, the product having a satisfiable water-dispersibility and taste preference can be advantageously used as a food product for health, antibacterial agent and agent for urine therapy in the maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

[Example 6]

Intact propolis was dewaxed with ethyl ether and extracted with a relatively-high concentration of an aqueous ethanol solution to obtain a dewaxed propolis extract having about 74 w/w % ethanol, about 8 w/w % moisture and about 18 w/w % propolis components. Five parts by weight of the dewaxed propolis extract thus obtained was admixed to homogeneity with one part by weight of a ginseng extract hydrate and 10 parts by weight of an anhydrous crystalline glucose powder, and the resultant mixture was allowed to stand at ambient temperature for 3 hours, and further admixed to homogeneity with 5 parts by weight of an anhydrous crystalline glucose powder to obtain a solid product in powder. One g aliquots of the solid product were injected in laminated aluminum-bags and sealed.

Similarly as the product in Example 1, the product having a satisfiable water-dispersibility and taste preference can be advantageously used as a food product for health, antibacterial agent and agent for urine therapy in the maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

[Effect of the invention]

As evident from above, the solid product according to the present invention is prepared by incorporating propolis components, the main components of propolis, which are hydrophobic or scarcely soluble in water but soluble in a readily water-soluble organic-solvent, in one or more saccharides selected from anhydrous saccharides and cyclodextrins. The solid product thus obtained has a satisfiable water-dispersibility and taste preference, and can overcome the drawbacks of conventional propolis extracts in liquid and other propolis products in solid wherein emulsifiers or surfactants are inevitably used.

The present solid product, similarly as conventional propolis extracts, can be advantageously used as a food product for health, antibacterial agent and therapeutic agent in the maintenance and promotion of health, and the prevention and treatment of diseases, as well as in the promotion of recovery of health from diseases.

It was also found that the present solid product can

be advantageously used as a flavor-imparting agent, deodorant, sublingual agent and agent for urine therapy.

CLAIMS:

1. A readily water-dispersible solid product which comprises a saccharide(s) and propolis components, which are contained in intact propolis or dewaxed propolis preparations and soluble in an aqueous solution of an organic solvent, said product being prepared by:

(a) selecting a quantity of material selected from the group consisting of intact propolis and dewaxed propolis preparations;

(b) extracting said quantity of material with an aqueous solution of an organic solvent selected from the group consisting of acetone, acetic acid and alcohols, wherein the concentration of said organic solvent in said aqueous solution is 30 w/w % or higher to form a first extract containing propolis components which are contained in said quantity of material and soluble in said organic solvent;

(c) evaporating water from the first extract containing said propolis components to give a moisture content of 1 w/w % or higher but lower than 50 w/w % to form a second extract;

(d) incorporating the second extract, which contains said propolis components, in one or more saccharides selected from the group consisting of anhydrous saccharides and cyclodextrins in order to dehydrate the second extract, wherein the amount of said extract against that of said saccharide(s) is in the range of 0.1-50 w/w %, on a dry solid basis;

(e) drying the dehydrated second extract in the step (d) at ambient temperature; and

(f) incorporating the resultant extract in the step (e) in an effective amount of one or more saccharides selected from the group consisting of anhydrous

saccharides and cyclodextrins to obtain a water-dispersible solid product.

2. A solid product according to claim 1, wherein the step (f) further contains a step of mixing said solid product in the step (f) with urine.

3. A solid product according to claim 1 or claim 2, wherein said anhydrous saccharide is an anhydrous crystalline saccharide.

4. A solid product according to claim 3, wherein said anhydrous crystalline saccharide is a member selected from the group consisting of anhydrous maltose, lactitol, glucose, galactose, paratinose, raffinose, erlose and melezitose.

5. A solid product according to any one of the preceding claims, wherein said organic-solvent is a member selected from the group consisting of methanol, ethanol and propanol.

6. A solid product according to any one of the preceding claims, wherein the moisture content of said saccharide is 3 w/w % or lower.

7. A solid product according to any one of the preceding claims, which is in the form of powder, granule or tablet.

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8. A solid product according to any one of the preceding claims, which is a food product for health, antibacterial agent, therapeutic agent, sublingual agent or agent for urine therapy.

9. A solid product according to claim 8, a dose of which is in the range of about 0.01-5g/day/adult, based on the weight of propolis components.

10. A process for preparing a readily water-dispersible solid product which comprises a saccharide(s) and propolis components, which are contained in intact propolis or dewaxed propolis preparations and soluble in an aqueous solution of an organic solvent, said process comprising:

(a) selecting a quantity of material selected from the group consisting of intact propolis and dewaxed propolis preparations;

(b) extracting said quantity of material with an aqueous solution of an organic solvent selected from the group consisting of acetone, acetic acid and alcohols, wherein the concentration of said organic solvent in said aqueous solution is 30 w/w % or higher to form a first extract containing propolis components which are contained in said quantity of material and soluble in said organic solvent;

(c) evaporating water from the first extract containing said propolis components to give a moisture content of 1 w/w % or higher but lower than 50 w/w % to form a second extract;

(d) incorporating the second extract, which contains said propolis components, in one or more saccharides selected from the group consisting of

anhydrous saccharides and cyclodextrins in order to dehydrate the second extract, wherein the amount of said extract against that of said saccharide(s) is in the range of 0.1-50 w/w %, on a dry solid basis;

(e) drying the dehydrated second extract in the step (d) at ambient temperature; and

(f) incorporating the resultant extract in the step (e) in an effective amount of one or more saccharides selected from the group consisting of anhydrous saccharides and cyclodextrins to obtain a water-dispersible solid product.

11. The process according claim 10, wherein said anhydrous saccharide is an anhydrous crystalline saccharide.

12. A process according to claim 11, wherein said anhydrous crystalline saccharide is a member selected from the group consisting of anhydrous maltose, lactitol, glucose, galactose, paratinose, raffinose, erlose and melezitose.

13. A process according to any one of claims 10 to 12, wherein said aqueous solution is a member selected from the group consisting of aqueous solutions of methanol, ethanol and propanol.

14. A process according to any one of claims 10 to 13, wherein the moisture content of said saccharide is 3 w/w % or lower.

15. A process according to any one of claims 10 to 14, wherein the solid product is in the form of powder, granule or tablet.

16. A process according to any one of claims 10 to 15, wherein said solid product is a food product for health, antibacterial agent, therapeutic agent, sublingual agent or agent for urine therapy.

17. A process according to claim 16, wherein a dose of said solid product is in the range of about 0.01-5g/day/adult, based on the weight of propolis components.

18. A process for making a solid product according to claim 10 and substantially as hereinbefore described with reference to any one of the Examples.

19. A solid product obtainable by the process of any one of claims 10 to 18.

20. A solid product according to claim 1, substantially as hereinbefore described.