# LYCOPENE MICROCAPSULE PREPARATION METHOD

## BACKGROUND

### Technical Field

The present invention relates to the technical field of traditional Chinese medicine, and in particular to a lycopene microcapsule preparation method.

### Related Art

Lycopene, a type of carotenoids, is widely found in plants such as tomatoes, watermelons, grapes, and carrots. Due to lack in biological activity of vitamin A, effects of the lycopene attracted less attention. However, studies in recent years indicated that, the lycopene has more excellent performance than other carotenes. For example, capability of the lycopene in removing singlet oxygen is 100 times that of vitamin E, and more than 2 times that of β-carotene. The lycopene is capable of improving immunity of organisms, restraining multiplication of cancer cells, reducing loss of DNA, preventing oxidation of low-density lipoprotein, and lowering content of blood cholesterol, thus reducing incidence of many diseases.

Generally, the lycopene exists in a form of deep red powder or oily liquid. Pure lycopene is a needle-shaped deep red crystal, which turns orange yellow in low concentration solutions, with melting point of 174°C (trans-form), and is flammable. As an aliphatic hydrocarbon, the lycopene is insoluble in water, and hardly soluble in polar organic solvents such as methanol and ethanol. However, the lycopene is soluble in diethyl ether, petroleum ether, ethane, and acetone, and easily soluble in chloroform, carbon disulfide, benzene, grease and the like. The higher purity the lycopene has, the more difficultly it dissolves. Solubility of the lycopene in various solvents will increase as temperature rises. The lycopene contains abundant unsaturated structures, and is easily degraded and isomerized due to effect of factors such as heat, light, oxygen and additives during processing and storage, thus reducing the biological activity.

In order to fully utilize the lycopene, a micro-encapsulation technology is adopted to

encapsulate the lycopene, preventing the lycopene from directly contacting external environment, and thus improving stability. The micro-encapsulation technology refers to encapsulating a liquid, solid or even gas (a core material) with a suitable film forming material (a wall material) to form micro-particles. After micro-encapsulation processing, the core material does not directly contact external environmental factors, thereby being effectively protected. However, the prior art lycopene micro-encapsulation process is difficult to ensure water solubility of lycopene microcapsules and long-term stability of content of active ingredients thereof.

## SUMMARY

An objective of the present invention is to provide a lycopene microcapsule preparation method, which improves stability and water solubility, thus expanding the application scope of the lycopene microcapsules.

The objective of the present invention is achieved by the following manner:

A lycopene microcapsule preparation method mainly includes the steps of: firstly preparing lycopene into a core material, and then homogeneously emulsifying and spray-drying the core material and a wall material, where the wall material is prepared with a method as follows: using gelatin, sporoderm-broken ganoderma lucidum spore powder, cane sugar and skim milk powder in a weight ratio of 1:1:1:1.2-1.6 as starting materials of the wall material, adding distilled water 3-5 times the quantity of the above materials, heating to 40-45°C and stirring to form a homogeneous suspension as the wall material. Preferably, the wall material is prepared with a method as follows: using the gelatin, the sporoderm-broken ganoderma lucidum spore powder, the cane sugar and the skim milk powder in a weight ratio of 1:1:1:1.4 as the starting materials of the wall material, adding the distilled water 4 times the quantity of the above materials, heating to 45°C and stirring to form a homogeneous suspension as the wall material.

The core material is prepared with a method as follows: dissolving every 0.5-0.8 g lycopene in 1 ml salad oil, adding sucrose ester as an emulsifying agent, and stirring evenly to form the core material, where a weight ratio of the lycopene to the sucrose ester is 10-14:1.

Preferably, the core material is prepared with a method as follows: dissolving every 0.68 g lycopene in 1 ml salad oil, adding the sucrose ester as the emulsifying agent, and stirring evenly to form the core material, where the weight ratio of the lycopene to the sucrose ester is 8.5:0.7.

The salad oil is a common edible salad oil, such as soybean salad oil, rapeseed salad oil, sunflower seed salad oil, and rice bran salad oil. The sporoderm-broken ganoderma lucidum spore powder may preferably be sporoderm-broken ganoderma lucidum spore powder with a sporoderm-broken rate greater than 95%.

The homogeneously emulsifying includes the following steps: using a mixed solution of the core material and the wall material as an oil phase, stirring water and slowly adding the oil phase into the water, where a weight ratio of the water to the oil phase is 3-3.5:5-6, and then placing the water and the oil phase into a colloid mill for grinding at 5,000-10,000 rpm over 20-40 min.

Preferably, the homogeneously emulsifying includes the following steps: using the mixed solution of the core material and the wall material as the oil phase, stirring the water and slowly adding the oil phase into the water, where the weight ratio of the water to the oil phase is 3:5, and then placing the water and the oil phase into the colloid mill for grinding at 6,000 rpm over 30 min.

The spray-drying is conducted under conditions as follows: during the spray-drying, an inlet air temperature is 170-180°C, an outlet air temperature is 80-100°C, a charging temperature 45-50°C, a charging rate is 0.6-1.0 kg/min, and a blowing rate is 50-60 L/min. Preferably, the spray-drying is conducted under conditions as follows: during the spray-drying, the inlet air temperature is 180°C, the outlet air temperature is 80-100°C, the charging temperature is 50°C, the charging rate is 0.6-1.0 kg/min, and the blowing rate is 50-60 L/min.

Compared with the prior art, the present invention has the following beneficial effects:

The present invention adopts a film-forming material that conforms to national requirements for food additives, causes no toxicity to human body, and easily releases the core material when exposed to water as the wall material. Additionally, the present invention

uses an appropriate micro-encapsulation method to encapsulate the lycopene within a micro-sized and enclosed film, transforming the lycopene to solid powder that can be easily processed, so as to protect the sensitive lycopene against oxidation. Particularly, the wall material adopted in the present invention is made from new materials, and is capable of achieving slow release of the core material, prolonging acting time of functional factors, and significantly improving the stability of lycopene microcapsules. Moreover, the core material prepared with formula of the present invention enables the wall material used in the present invention to easily achieve encapsulation. Specific micro-encapsulation technology adopted in the present invention to encapsulate the lycopene, may improve stability of active ingredients, and also improve the water solubility of lycopene microcapsules, thus expanding the scope of the application.

## DETAILED DESCRIPTION

The present invention will be further described with reference to embodiments in the following. Specific implementations of the following embodiments merely illustrate the present invention in more details, but the present invention is not limited to content of the following embodiments. As described in the following embodiments, lycopene refers to the lycopene raw material produced by Lycored Corp., Israel, with a purity of 98%; salad oil refers to Jinlongyu soybean salad oil manufactured by Shanghai Kerry Oils & Grains Industries Co., Ltd.; sucrose ester (i.e. sucrose fatty acid ester) is produced by Adana Food Tech (Liuzhou) Co., Ltd.; sporoderm-broken ganoderma lucidum spore powder (with a sporoderm-broken rate greater than 95%) is manufactured by Nanjing Zhongke Pharmacy Co., Ltd.; skim milk powder is manufactured by Heilongjiang Feihe Dairy Co., Ltd.; and lycopene microcapsules 1 purchased from market are produced by Parry Nutraceuticals Limited, India, and lycopene microcapsules 2 purchased from market are manufactured by Lycored Corp., Israel.

### Embodiment 1

1. Preparation of a core material: 8.5 g lycopene was added into 12.5 ml salad oil, and 0.7 g sucrose ester was added as an emulsifying agent. After stirring evenly, an oil phase A was formed.

2. Preparation of a wall material: gelatin, sporoderm-broken ganoderma lucidum spore powder, cane sugar and skim milk powder in a weight ratio of 1:1:1:1.4 were taken as starting materials of the wall material, and distilled water 4 times the quantity of the starting materials of the wall material was added therein. After heating to 45°C and stirring, a homogeneous suspension was formed as an oil phase B.

3. Homogeneously emulsifying: while stirring the water, a mixed solution of the oil phase A and the oil phase B was slowly added, in which a weight ratio of the water to oil phase (i.e. the sum of the oil phase A and the oil phase B) was 3:5. The solution was then placed into a colloid mill and grinded at 6,000 rpm over 30 min, to disperse the lycopene evenly in the wall material, thus obtaining a homogeneous emulsion.

The homogeneous emulsion was spray-dried to yield powdery lycopene microcapsules. The spray-drying was controlled under conditions as follows: during the spray-drying, an inlet air temperature was 180°C, an outlet air temperature 90°C, a charging temperature was 50°C, a charging rate was 0.8 kg/min and a blowing rate was 50-60 L/min.

In this embodiment of the present invention, a stability test for content of the lycopene microcapsules and results of the test are described as below:

The prepared lycopene microcapsules, non-microencapsulated lycopene raw material, lycopene microcapsules 1 purchased from market, and lycopene microcapsules 2 purchased from market, were separately charged in a transparent sample bottle and sealed for storage. Under irradiation by natural light at room temperature (20±5°C), samples were taken at regular intervals to determine content of the lycopene in the samples, and a retention rate was obtained by comparison to content of the lycopene in the samples prior to storage. Table 1 shows the results of the test for the content of the lycopene in different samples, in which the content of the lycopene was determined with a method as specified in the National Standard GB/T22249-2008.

Table 1

| Time / Samples | Starting time | 1 week | 2 weeks | 4 weeks | 24 weeks | 48 weeks | 96 weeks |
|---|---|---|---|---|---|---|---|
| Lycopene raw material | 98% | 20% | 3.5% | 0 | 0 | 0 | 0 |
| Lycopene microcapsules in this embodiment | 20% | 20.2% | 20.1% | 20.0% | 19.6% | 18.9% | 17.2% |
| Lycopene microcapsules 1 purchased from market | 20% | 19.8% | 19.6% | 17% | 12.5% | 10.6% | 8.2% |
| Lycopene microcapsules 2 purchased from market | 20% | 19.6% | 19.2% | 16.3% | 11.4% | 8.9% | 7.0% |

In this embodiment of the present invention, results of tests for time limit of disintegration of the lycopene microcapsules are shown in Table 2. Three parallel tests were conducted with a method as specified in appendixes of the *Pharmacopoeia of the People's Republic of China* (2010 version).

Table 2

| | |
|---|---|
| Lycopene microcapsules in this embodiment | 46 min, 46 min, 47 min |
| Lycopene microcapsules 1 purchased from market | 92 min, 93 min, 95 min |
| Lycopene microcapsules 2 purchased from market | 103 min, 104 min, 102 min |

**Embodiment 2**

1. Preparation of a core material: 8 g lycopene was added into 12.5 ml salad oil, and 0.7 g sucrose ester was added as an emulsifying agent. After stirring evenly, an oil phase A was formed.

2. Preparation of a wall material: gelatin, sporoderm-broken ganoderma lucidum spore powder, cane sugar and skim milk powder in a weight ratio of 1:1:1:1.2 were taken as starting materials of the wall material, and distilled water 3 times the quantity of the starting materials

of the wall material was added therein. After heating to 45°C and stirring, a homogeneous suspension was formed as an oil phase B.

3. Homogeneously emulsifying: while stirring the water, a mixed solution of the oil phase A and the oil phase B was slowly added, in which a weight ratio of the water to oil phase (i.e. the sum of the oil phase A and the oil phase B) was 3:5. The solution was then placed into a colloid mill and grinded at 8,000 rpm over 25 min, to disperse the lycopene evenly in the wall material, thus obtaining a homogeneous emulsion.

The homogeneous emulsion was spray-dried to yield powdery lycopene products. The spray-drying was controlled under conditions as follows: during the spray-drying, an inlet air temperature was 180°C, an outlet air temperature was 80°C, a charging temperature was 45°C, a charging rate was 0.6 kg/min and a blowing rate was 50-60 L/min.

In this embodiment of the present invention, results of a stability test for content of the lycopene in the lycopene microcapsules are shown in Table 3, with the same test method as Embodiment 1.

Table 3

| Time / Samples | Starting time | 1 week | 2 weeks | 4 weeks | 24 weeks | 48 weeks | 96 weeks |
|---|---|---|---|---|---|---|---|
| Raw materials of lycopene | 98% | 20% | 3.5% | 0 | 0 | 0 | 0 |
| Lycopene microcapsules in this embodiment | 20% | 20% | 20% | 19.8% | 19.4% | 18.7% | 17% |

In this embodiment of the present invention, tests of time limit for disintegration of the lycopene microcapsules were conducted with a method as specified in appendixes of the *Pharmacopoeia of the People's Republic of China* (2010 version). Average disintegration time determined was 46 min through 3 parallel tests.

**Embodiment 3**

1. Preparation of a core material: 9 g lycopene was added into 12.5 ml salad oil, and 0.7

g sucrose ester was added as an emulsifying agent. After stirring evenly, an oil phase A was formed.

2. Preparation of a wall material: gelatin, sporoderm-broken ganoderma lucidum spore powder, cane sugar and skim milk powder in a weight ratio of 1:1:1:1.6 were taken as starting materials of the wall material, and distilled water 5 times the quantity of the starting materials of the wall material was added therein. After heating to 40°C and stirring, a homogeneous suspension was formed as an oil phase B.

3. Homogeneously emulsifying: while stirring the water, a mixed solution of the oil phase A and the oil phase B was slowly added, in which a weight ratio of the water to oil phase (i.e. the sum of the oil phase A and the oil phase B) was 3.5:5. The solution was then placed into a colloid mill and grinded at 5,000 rpm over 35 min, to disperse the lycopene evenly in the wall material, thus obtaining a homogeneous emulsion.

The homogeneous emulsion was spray-dried to yield powdery lycopene products. The spray-drying was controlled under conditions as follows: during the spray-drying, an inlet air temperature was 170°C, an outlet air temperature was 100°C, a charging temperature was 50°C, a charging rate was 1.0 kg/min and a blowing rate was 50-60 L/min.

In this embodiment of the present invention, results of a stability test for content of the lycopene in the lycopene microcapsules are shown in Table 4, with the same test method as Embodiment 1.

Table 4

| Time / Samples | Starting time | 1 week | 2 weeks | 4 weeks | 24 weeks | 48 weeks | 96 weeks |
|---|---|---|---|---|---|---|---|
| Raw materials of lycopene | 98% | 20% | 3.5% | 0 | 0 | 0 | 0 |
| Lycopene microcapsules in this embodiment | 20% | 20.1% | 20% | 19.7% | 19.3% | 18.5% | 16.9% |

In this embodiment of the present invention, tests of time limit for disintegration of the

lycopene microcapsules were conducted with a method as specified in appendixes of the *Pharmacopoeia of the People's Republic of China* (2010 version). Average disintegration time determined was 48 min through 3 parallel tests.

## CLAIMS

What is claimed is:

1. A lycopene microcapsule preparation method, mainly comprising: firstly preparing lycopene into a core material, and then homogeneously emulsifying and spray-drying the core material and a wall material, wherein the wall material is prepared with a method as follows: using gelatin, sporoderm-broken ganoderma lucidum spore powder, cane sugar and skim milk powder in a weight ratio of 1:1:1:1.2-1.6 as starting materials of the wall material, adding distilled water 3-5 times the quantity of the above materials, heating to 40-45°C and stirring to form a homogeneous suspension as the wall material.

2. The lycopene microcapsule preparation method according to claim 1, wherein the wall material is prepared with a method as follows: using the gelatin, the sporoderm-broken ganoderma lucidum spore powder, the cane sugar and the skim milk powder in the weight ratio of 1:1:1:1.4 as the starting materials of the wall material, adding the distilled water 4 times the quantity of the above materials, heating to 45°C and stirring to form a homogeneous suspension as the wall material.

3. The lycopene microcapsule preparation method according to claim 1, wherein the core material is prepared with a method as follows: dissolving every 0.5-0.8 g lycopene in 1 ml salad oil, and adding sucrose ester as an emulsifying agent to form the core material, wherein a weight ratio of the lycopene to the sucrose ester is 10-14:1.

4. The lycopene microcapsule preparation method according to claim 3, wherein the core material is prepared with a method as follows: dissolving every 0.68 g lycopene in 1 ml salad oil, and adding the sucrose ester as the emulsifying agent to form the core material, wherein the weight ratio of the lycopene to the sucrose ester is 8.5:0.7.

5. The lycopene microcapsule preparation method according to claim 1, wherein the homogeneously emulsifying comprises the following steps: using a mixed solution of the core material and the wall material as an oil phase, stirring water and slowly adding the oil phase into the water, wherein a weight ratio of the water to the oil phase is 3-3.5:5-6, and then placing the water and the oil phase into a colloid mill for grinding at 5,000-10,000 rpm

over 20-40 min.

6. The lycopene microcapsule preparation method according to claim 5, wherein the homogeneously emulsifying comprises the following steps: using the mixed solution of the core material and the wall material as the oil phase, stirring the water and slowly adding the oil phase into the water, wherein the weight ratio of the water to the oil phase is 3:5, and then placing the water and the oil phase into the colloid mill for grinding at 6,000 rpm over 30 min.

7. The lycopene microcapsule preparation method according to claim 1, wherein the spray-drying is conducted under conditions as follows: during the spray-drying, an inlet air temperature is 170-180°C, an outlet air temperature is 80-100°C, a charging temperature is 45-50°C, a charging rate is 0.6-1.0 kg/min and a blowing rate is 50-60 L/min.

8. The lycopene microcapsule preparation method according to claim 7, wherein the spray-drying is conducted under conditions as follows: during the spray-drying, the inlet air temperature is 180°C, the outlet air temperature is 80-100°C, the charging temperature is 50°C, the charging rate is 0.6-1.0 kg/min and the blowing rate is 50-60 L/min.