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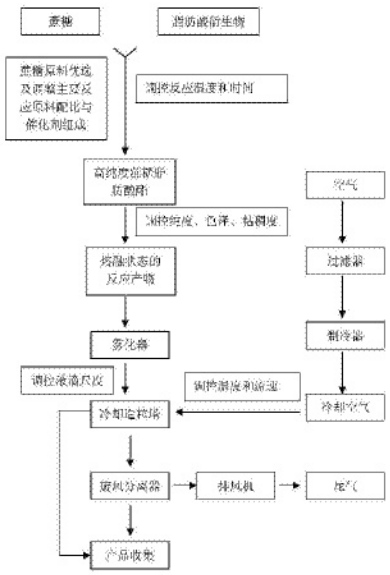
(54) Name of invention

Method for preparing sucrose fatty acid ester granules by spray cooling

(57) Abstract

The invention relates to a method for preparing sucrose fatty acid ester particles by spray cooling. The method is to atomize sucrose fatty acid ester in molten state produced by solvation-free method into a cooling tower directly through an atomizer, and then heat exchange with the air in the cooling tower and cure to obtain sucrose fatty acid ester granule finished product. The invention is completely different from the traditional production process of sucrose fatty acid ester, and the reaction product does not need to be cooled naturally or crushed by machinery. The whole production process of sucrose fatty acid ester from feeding, reaction to final product is completed at one time in a completely closed environment, avoiding the environmental pollution, product pollution and human harm that may be caused by dust, and greatly improving the quality of sucrose fatty acid ester products. Compared with the traditional powdered sucrose fatty acid ester processed by the traditional machinery, the technology has the advantages of uniform particle size distribution, high sphericity, good fluidity, not easy to be damp, high wettability and solubility.



CN 108067160 A

1. A method for preparing sucrose fatty acid ester particles by spray cooling, which is characterized in that: the molten sucrose fatty acid ester produced by solveless method is atomized and sprayed in the cooling granulation tower directly through the atomizer, and the cold air in the cooling granulation tower is heated and cooled and solidified to obtain sucrose fatty acid ester particles;

The solvent-free method of producing molten state of sucrose fatty acid ester specific conditions are: reaction raw material sucrose and fatty acid ester mass ratio of 1:1.4~6, the amount of catalyst for the total mass of raw materials 1%~5%, the whole reaction system should be controlled in vacuum degree greater than 0.094MPa vacuum state, reaction temperature should be controlled at 120℃~140℃, reaction Time should be controlled in 3~ 6 hours,

The atomizer spray cooling conditions are: the atomizer speed is 2000~12000r/min, the inlet air temperature is 5~20℃, the outlet air temperature is 30~45℃, the feed temperature is 100~130℃, the feed speed is 30~600L/h, and the cold air flow rate is 100~1000 cubic meters/hour.

2. According to the method of preparing sucrose fatty acid ester particles by spray cooling described in claim 1, it is characterized by: the mass ratio of sucrose and fatty acid ester as reaction raw materials is 1:4, and the amount of catalyst is 3.5% of the total mass of raw materials.

3. According to a method of spray cooling for preparing sucrose fatty acid ester particles described in claim 1 or 2, its characteristics are: the reaction raw material sucrose is refined white granulated sugar, and the reducing sugar content should be less than 0.2%.

4. According to claim 1 or 2 of a method of spray cooling preparation of sucrose fatty acid ester particles, which is characterized by: sucrose fatty acid ester particles finished with a particle size of 10~500um.

· A method of spray cooling for preparing sucrose fatty acid ester particles in accordance with claim 1 or 2 is characterized in that the catalyst is food-grade potassium carbonate or/and food-grade potassium hydroxide.

A method for preparing sucrose fatty acid ester particles by spray cooling

Technical field

[0001 The invention relates to a method for sucrose fatty acid ester granules, in particular to a method for preparing sucrose fatty acid ester granules by spray cooling.

Background technology

[0002] As the main production method for sucrose fatty acid ester is solvent-free method, and the solvent-free sucrose fatty acid ester production rule is to esterify sucrose and fatty acid ester in the molten phase by high temperature, so the traditional sucrose fatty acid ester reaction product needs to be cooled naturally into block or waxy solid, and then processed into powder by mechanical grinding Finished product. In addition, due to the moisture absorption property of sucrose itself, especially the excessive reducing sugar content in the sucrose fatty acid ester reaction products in the process of natural cooling and mechanical crushing, as well as the storage process of finished products, sucrose fatty acid ester products are easy to moisture absorption agglomeration; Therefore, the traditional solvent-free sucrose fatty acid ester production process, especially the post-processing process, has seriously affected the appearance quality and performance of sucrose fatty acid ester products. In addition, the traditional mechanical grinding process also produces a lot of dust and may cause pollution to the environment and products, and may also affect the health of the direct production personnel.

Content of the invention

[0003] The technical problem to be solved is that the invention provides a method for preparing sucrose fatty acid ester particles by spray cooling. The reaction product of the method does not need natural cooling or mechanical crushing, so that the production of sucrose fatty acid ester from feeding, reaction to the final product can be completed at one time in a completely closed environment to avoid environmental pollution, product pollution, dust pollution, etc. Harm to human body, etc., and greatly improve the quality of sucrose fatty acid ester products.

[0004] The technical solution to the above technical problems is as follows: a method for preparing sucrose fatty acid ester particles by spray cooling. The molten sucrose fatty acid ester produced by solvless method is atomized and sprayed in the cooling granulation tower directly through the atomizer, and the cold air in the cooling granulation tower is exchanged for heat and then cooled and solidified to obtain sucrose fatty acid ester particles;

The specific conditions of the solvent-free production of molten state of sucrose fatty acid ester are: the mass ratio of raw material sucrose and fatty acid ester is 1:1.4~6, the amount of catalyst is 1%~5% of the total mass of raw material, the whole reaction system should be controlled in the vacuum degree greater than 0.094MPa vacuum state, the reaction temperature should be controlled at 120℃~140℃, the reaction Time should be controlled in 3~ 6 hours,

The atomizer spray cooling conditions are: the atomizer speed is 2000~12000r/min, the inlet air temperature is 5~20℃, the outlet air temperature is 30~45℃, the feed temperature is 100~130℃, the feed speed is 30~600L/h, and the cold air flow rate is 100~1000 cubic meters/hour.

[0005] The further technical features of the invention are that the mass ratio of sucrose and fatty acid ester of the reaction raw material is 1:4, and the amount of catalyst is 3.5% of the total mass of the raw material.

[0006 The reaction raw material sucrose is refined white granulated sugar, and the reducing sugar content should be less than 0.2%.

[0007] The particle size of the finished sucrose fatty acid ester granule is 10-500um.

[0008] The catalyst is food grade potassium carbonate or/and food grade potassium hydroxide.

[000 hot melt spray cooling technology, without the use of any solvent, the molten material is dispersed into mist droplets through the atomizer and in the cooling room and cold air in full contact for heat exchange, so that the mist droplets quickly solidified in the high efficiency cyclone separator

A special preparation means to complete gas-solid separation to obtain very fine powder or fine particle semi-finished product or finished product with uniform finness and quality, which can be used for revolutionary improvement of the traditional sucrose fatty acid ester production process. That is, sucrose fatty acid ester granule products are prepared by hot melt spray cooling technology, which can improve the particle size distribution, sphericity, fluidity, wettability and solubility of sucrose fatty acid ester products, and improve product quality.

[0010] Sucrose fatty acid ester is generated by transesterification reaction between sucrose and fatty acid ester in the melt phase through high temperature, and the reaction temperature is between 120℃ and 140℃, which is easy to coking and discoloration, making the reaction product show a yellowish viscous or resinous liquid form, deviating from the normal colorless or white state. In addition, the traditional mechanical grinding process also produces a large amount of dust, which may pollute the environment and the product, and may also affect the health of the direct production personnel. Therefore, the preparation of sucrose fatty acid ester granule products by hot melt spray cooling can not only give sucrose fatty acid ester products uniform particle size distribution, high sphericity, good fluidity, high wettability and solubility, improve product quality, but also make sucrose fatty acid ester production from feeding, reaction to the final product of the entire production process in a completely closed environment to complete at one time, avoid Dust may cause environmental pollution, product pollution, human harm and so on.

[0011] The beneficial effects of the invention are described as follows:

1. Sucrose fatty acid ester production, from feeding, reaction to the final product, the whole production process is completed at one time in a completely closed environment. Compared with the traditional production process of sucrose fatty acid ester, the production efficiency and product quality have been improved.

[0012] 2· Compared with the traditional powdered sucrose fatty acid ester processed by traditional mechanical grinding, this technology has the advantages of uniform particle size distribution, high sphericity, good fluidity, not easy to be damp, good wettability, etc. The original physical and chemical properties of the product can be effectively maintained, and the product is easy to be stored for a long time.

[0013] Due to the excellent wettability of the technology, the product can be quickly dispersed and dissolved, so it can avoid the traditional powdered sucrose fatty acid ester products which are difficult to disperse and dissolve, and even the occurrence of clumping.

[0014] 4· Sucrose fatty acid ester production without mechanical crushing, to avoid dust may cause environmental pollution, product pollution, human harm and so on.

Illustrated description

[0015]

Figure 1: Process flow diagram of the invention.

[0016] Figure 2: Microscope magnification of sucrose fatty acid ester particles prepared by the invention (smaller magnification)

[0017] Figure 3: Microscope magnification diagram of sucrose fatty acid ester particles prepared by the invention (larger magnification).

Specific embodiments

[0018] Embodiments 1

1) Sucrose is sulfite process mechanism white granulated sugar, reducing sugar content is 0.50%.

[0019] 2) The ratio of white granulated sugar and fatty acid methyl ester as the main reaction raw material of sucrose fatty acid ester is 1:4, and the proportion of catalyst composition is 3.5% of the total mass of raw material (that is, 3.5% of the total mass of white granulated sugar and fatty acid methyl ester). The whole reaction system should be controlled in vacuum state, and the vacuum degree should be kept at 0.098 MPa, the reaction temperature is controlled at 125±1℃, and the reaction time is controlled at 3 hours.

[0020] The molten state of sucrose fatty acid ester produced by solvent-free method should maintain a good molten state, the material temperature is 125±1℃, and maintain fluidity.

[0021] 3) The high temperature solution of sucrose fatty acid ester after the reaction is passed into the centrifugal atomizer for spray cooling. The conditions of spray cooling are as follows: centrifugal disc rotation speed of 7000r/min, inlet air temperature of 16℃, outlet air temperature of 30-45℃, and feed temperature

At 123℃, the feed speed is 60L/h, and the cold air flow is controlled at 200m/h. The product obtained by using this atomization condition is usually regular spherical particles with excellent fluidity.

[0022] The final product of sucrose fatty acid ester obtained by this method is a particle size of 200~400um. Due to excessive reducing sugar, the material is light brown and the color is not ideal.

[0023] Embodiments 2

1) Sucrose is the carbonic acid mechanism sugar white granulated sugar, the reducing sugar content is 0.03%.

[0024] 2) The main reaction raw material of sucrose fatty acid ester is white granulated sugar and fatty acid methyl ester ratio is 1:4, the catalyst composition ratio is 3.5%, the whole reaction system should be controlled in a vacuum state, the vacuum degree is maintained at 0.098MPa, the reaction temperature is controlled at 123±1℃, and the reaction time is controlled at 3 hours.

[0025] The molten sucrose fatty acid ester produced by solvent-free method maintained a good molten state, the material temperature was 123±1℃, and the fluidity was good.

[0026] 3) The high temperature solution of sucrose fatty acid ester was passed into the centrifugal atomizer for spray cooling. The spray cooling conditions were as follows :7000r/min, the inlet air temperature was 16℃, the outlet air temperature varied between 30-45℃, the inlet temperature was 123℃, the inlet speed was 60L/h, and the cold air flow was controlled at 200m/h. The product obtained by using this atomization condition is usually regular spherical particles with excellent fluidity.

[0027] The final product of sucrose fatty acid ester obtained by this method has a particle size of 86% ranging from 200 to 500m. Due to the extremely low reducing sugar content, it is pure white and has good color.

[0028] Embodiment 3

1) Sucrose is white granulated sugar by carbonation process, and the reducing sugar content is 0.03%.

[0029] 2) The main reaction raw material of sucrose fatty acid ester is white granulated sugar and fatty acid methyl ester ratio is 1:4, the catalyst composition ratio is 3.5%, the whole reaction system should be controlled in vacuum state, the vacuum degree is maintained at 0.098MPa, the reaction temperature is controlled at 123±1℃, and the reaction time is controlled at 3 hours.

[0030] The molten state of sucrose fatty acid ester produced by solvent-free method maintains a good molten state, and the temperature of the material is 123±1℃, and the fluidity is maintained.

3) The high temperature solution of sucrose fatty acid ester reaction product is passed into the centrifugal atomizer for cooling spray, the spray cooling condition is :9000r/min, the inlet air temperature is 16℃, the outlet air temperature is varied between 30-45℃, the feed temperature is 123℃, the feed speed is 60L/h, and the cold air flow is controlled by 200m/h. The product obtained by the atomization conditions is usually a regular spherical particle, with good fluidity.

[0031] The final product of sucrose fatty acid ester obtained by this method has a particle size of 92% ranging from 100 to 500m, which is pure white and has good color because it contains very low reducing sugar.

[0032] The catalyst of the invention is food-grade potassium carbonate and/or food-grade potassium hydroxide.

[0033] In various embodiments of the invention, the product obtained after atomization by the atomizer is sprayed in the cooling granulation tower, and the cold air in the cooling granulation tower is heat exchanged and cooled to obtain sucrose fatty acid ester particles. Most of the materials enter the silo at the bottom of the granulation tower by natural sedimentation, and a small amount of very fine materials enter the silo after separation by a cyclone separator.

[0034] In the embodiments of the invention, the size of the droplets in the atomizer is determined by the size of the centrifugal atomizing disc and the speed of the atomizing disc. The faster the speed, the finer the droplets. The general requirements of the process conditions of the cooling granulation tower are as following: the adjustment of the speed of the centrifugal disc of the cooling granulation tower is determined by the requirements of the fineness of the product. The finer the product requirements, the higher the speed; The adjustment of the amount of cold air is determined by the amount of feed, and the larger the amount of feed, the greater the amount of cold air required.

[0035] Table 1: Size distribution table of sucrose fatty acid ester particles obtained at different speeds of atomizer.

粒径（μm） / 转速（rpm）	10~100	100~200	200~300	300~400	400~500	500~600
7000		6%	30%	40%	16%	8%
9000	8%	18%	38%	22%	14%	

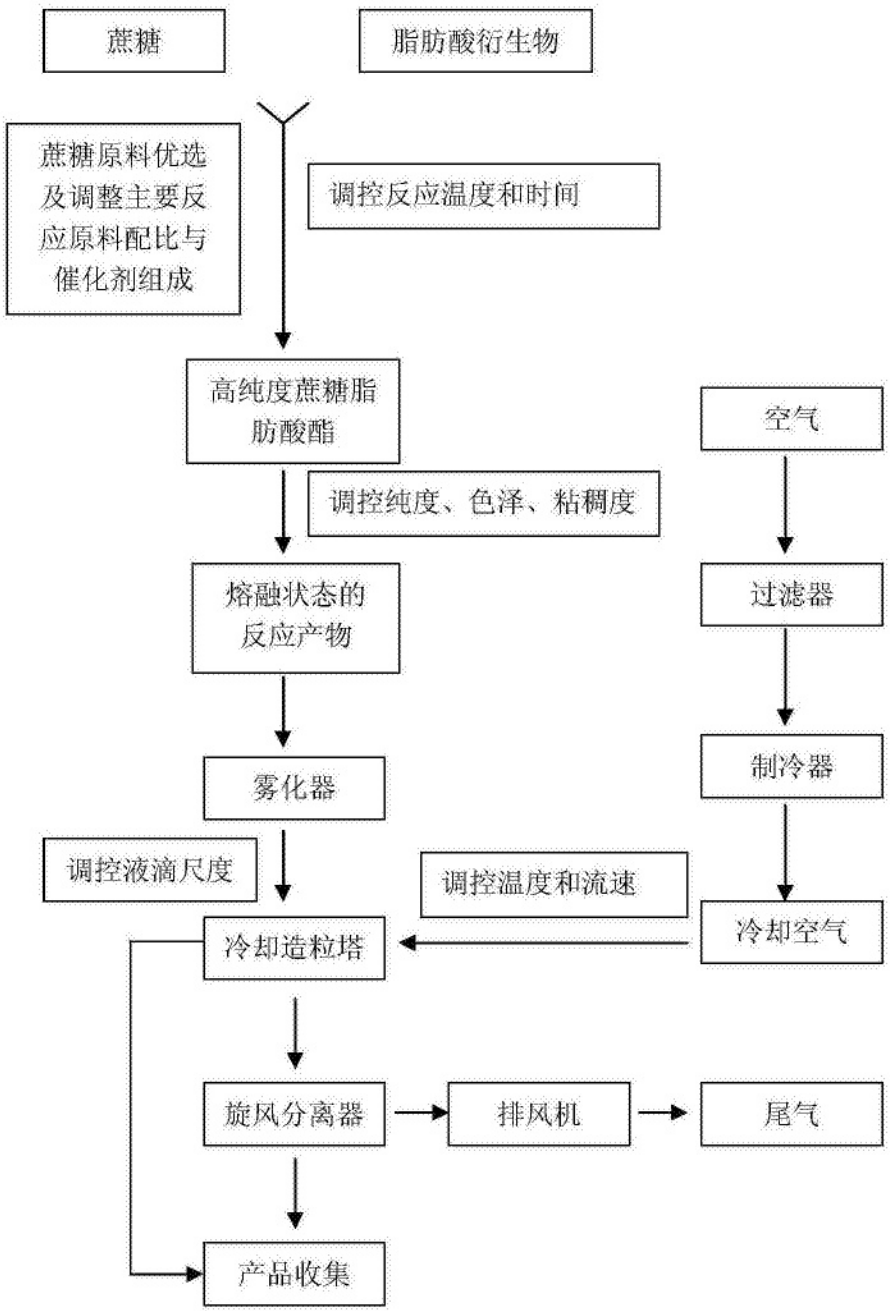

蔗糖
脂肪酸衍生物
蔗糖原料优选及调整主要反应原料配比与催化剂组成
调控反应温度和时间
高纯度蔗糖脂肪酸酯
空气
调控纯度、色泽、粘稠度
熔融状态的反应产物
过滤器
雾化器
制冷器
调控液滴尺度
调控温度和流速
冷却造粒塔
冷却空气
旋风分离器
排风机
尾气
产品收集

FIG. 1

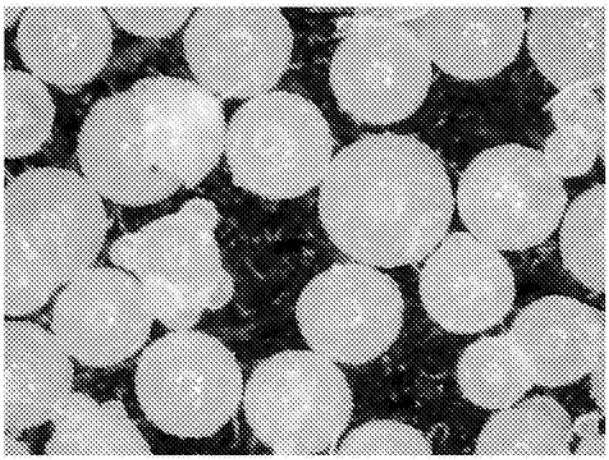


Figure 2

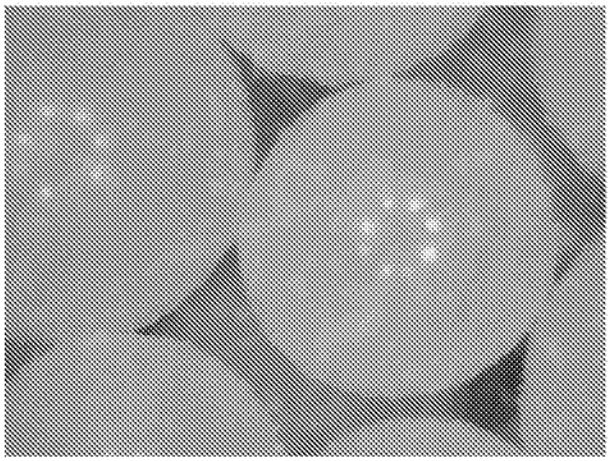


图3

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Claim 1 page note #3 with 2 pages of drawings

(54) Name of invention

- A method for preparing sucrose fatty acid ester granules integrated with drying molding

(57) Abstract

The invention relates to a preparation method of sucrose fatty acid ester particles integrated with drying and molding. The method first separates and purifies the sucrose fatty acid ester reaction product, and makes a liquid with a certain proportion of solid matter, and then atomizes it in a cooling tower through an atomizer. After heat exchange with the air in the cooling tower, the sucrose fatty acid ester particles are cooled and solidified, and finally the sucrose ester products are obtained by freeze-drying method to remove volatile components. Completely different from the traditional production process of sucrose fatty acid ester, the production of sucrose fatty acid ester involved in this method does not need to be dried and cooled first, and finally crushed, but after separation and purification, set drying and forming in one body, and completed in a completely closed environment through spray freeze-drying process to avoid environmental pollution, product pollution and human harm that may be caused by dust. At the same time, it greatly improves the quality of sucrose fatty acid ester products.

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1. A method for preparing sucrose fatty acid ester particles integrated with drying and forming is characterized in that: The sucrose fatty acid ester reaction products are separated and purified to obtain the sucrose fatty acid ester liquid with solid content greater than or equal to 85%. The sucrose fatty acid E liquid is sprayed through the atomizer to obtain granular sucrose fatty acid ester, and then sprayed in the cooling tower, and the air in the cooling tower is heated and solidified to obtain the sucrose fatty acid ester granule finished product;

The conditions of atomizer spray cooling are as follows: the atomizer speed is 6000~22000rpm, the inlet air temperature is 15~20℃, the outlet air temperature is 35~45℃, the feed temperature is 55~95℃, and the feed speed is 30~100L/h;

The freezing process in the cooling tower is: after the granular sucrose fatty acid ester is maintained at -40~-50℃ for 4~5h, the temperature rises to -28~-25℃ within 1.2-1.5h and is maintained at 28-30h; Then in 4~5h heating to -12~-10℃ and maintained for 4~5h; Then heating to 20~23℃ and maintained for 5~7h, the whole freezing process pressure is maintained at 0.01~100pa.

2. According to the preparation method of sucrose fatty acid ester particles described in claim 1, which is characterized by: the freezing process in the cooling tower is: the granular sucrose fatty acid ester is maintained at -45℃ for 5h, the temperature is raised to -25℃ and maintained for 30h within 1.5h, and then the temperature is raised to -10℃ and maintained for 5h within 5h, and then the temperature is raised to 20℃ And maintained for 7h.

3. According to claim 1 or 2, a preparation method of sucrose fatty acid ester particles integrated with drying and molding is characterized in that the prepared sucrose fatty acid ester particles have a particle size of 10~500m.

4. According to claim 1 or 2, a preparation method of sucrose fatty acid ester particles integrated with dry molding is characterized in that the atomizing disc diameter of the atomizer is 200 mm.

· According to claim 1 or 2, a method for preparing sucrose fatty acid ester particles integrated with drying and molding is characterized in that the separation and purification is carried out in accordance with the traditional method for the separation and purification of sucrose fatty acid ester, and the traditional method for the separation and purification of sucrose fatty acid ester is one or a combination of several methods of precipitation, aqueous solvent extraction and co-precipitation.

6. According to claim 1 or 2, a method for preparing sucrose fatty acid ester particles integrated with drying and molding is characterized in that the separation and purification is a molecular distillation method, a thin layer chromatography method or a liquid chromatography method.

- A method for preparing sucrose fatty acid ester particles integrated with dry molding

Technical field

[0001 The invention relates to a method for preparing sucrose fatty acid ester particles integrated with drying molding, in particular to a method for preparing sucrose fatty acid ester particles based on spray freeze drying technology.

Background technology

[0002] At present, there are four main methods for sucrose fatty acid ester synthesis, namely solvent method, microemulsification method, solvo-free method and enzyme catalysis method. No matter what method is used to produce sucrose fatty acid ester, sucrose fatty acid ester reaction products, either the content of the target product is not high, or a large number of impurities remain, and even some potentially toxic impurities remain. These impurities make sucrose fatty acid ester reaction products often can not meet the harsh requirements of food, drugs, cosmetics and other fields, can not be used directly, need further separation and purification, in order to expand the use of sucrose fatty acid ester fields and scope. In addition, due to the moisture absorption properties of sucrose itself, usually after separation and purification, sucrose fatty acid ester products also need to be dried, and then crushed to finally become the traditional powdered sucrose fatty acid ester finished products. Therefore, the traditional solvent-free sucrose fatty acid ester production process, especially the post-processing process, has seriously affected the appearance quality and performance of sucrose fatty acid ester products. In addition, the traditional mechanical grinding process also produces a lot of dust and may cause pollution to the environment and products, and may also affect the health of the direct production personnel.

Content of the invention

[0003] The technical problem to be solved is to provide a method for preparing sucrose fatty acid ester particles integrated with drying and forming. The method relates to the production of sucrose fatty acid ester without first drying, cooling and finally crushing, but after separation and purification, integrated drying and forming is completed in a completely closed environment through spray freeze-drying process to avoid possible dust Environmental pollution, product pollution, human harm, etc., while greatly improving the quality of sucrose fatty acid ester products.

[0004] The technical solution to the above technical problems is: a preparation method of sucrose fatty acid ester particles integrated with drying and molding, the separation and purification of sucrose fatty acid ester reaction products, to obtain a solid content of more than or equal to 85% sucrose fatty acid ester liquid, the sucrose fatty acid ester liquid through the atomizer spray cooling to obtain granular sucrose fatty acid ester, and then spray in the cooling tower. After heat exchange with the air in the cooling tower, the sucrose fatty acid ester particles are cured and finished;

The conditions of atomizer spray cooling are as follows: the atomizer speed is 6000~22000rpm, the inlet air temperature is 15~20℃, the outlet air temperature is 35~45℃, the feed temperature is 55~95℃, and the feed speed is 30~100L/h;

The freezing process in the cooling tower is: after the granular sucrose fatty acid ester is maintained at -40~-50℃ for 4~5h, the temperature rises to -28~-25℃ within 1.2-1.5h and is maintained at 28-30h; Then in 4~5h heating to -12~-10℃ and maintained for 4~5h; Then heating to 20~23℃ and maintained for 5~7h, the whole freezing process pressure is maintained at 0.01~100pa.

[0005] The further technical scheme of the invention is that the freezing process in the cooling tower is: after the granular sucrose fatty acid ester is maintained at -45 ℃ for 5h, it is heated to -25℃ within 1.5h and maintained for 30h, then it is heated to -10℃ within 5h and maintained for 5h, and then it is heated to 20℃ and maintained for 7h

[0006] The prepared sucrose fatty acid ester granule has a particle size of 10~500m

[0007] The atomizing disc of the atomizer has a diameter of 200 mm.

[0008] The separation and purification is carried out in accordance with the traditional sucrose fatty acid ester separation and purification method, the traditional sucrose lipid

The separation and purification method of fatty acid ester is one or a combination of several methods in precipitation method, aqueous solvent extraction method and co-precipitation method.

The separation and purification methods described in [000☒ are molecular distillation method, thin layer chromatography method or liquid chromatography method.

[0010] Spray freeze-drying technology is a combination of the advantages of spray drying and freeze-drying, not only effectively avoid the original shortcomings of spray drying and freeze-drying, but also, compared with freeze-drying technology, spray freeze-drying technology has lower energy consumption, in line with low carbon requirements, the dried sample is granular, the fluidity is very good, do not need to be re-crushed, compared with spray drying Spray freeze-drying has less damage to the active ingredients, and does not change the biological activity, so it is more widely used. Therefore, the invention just uses the spray freeze-drying technology to revolutionary improve the traditional production process of sucrose fatty acid ester, that is, the sucrose fatty acid ester reaction product is first separated and purified, and then the sucrose fatty acid ester is dried and granulated by spray freeze-drying technology, so as to improve the particle size distribution, sphericity, fluidity, wettability and solubility of the sucrose fatty acid ester product. Improve product quality, and avoid dust pollution to the environment and the product, which may affect the health of the direct production personnel.

[0011] The beneficial effects of the invention are described as follows:

1. It does not need to be dried and then crushed first, but after separation and purification, dry forming is integrated and completed at one time in a completely closed environment by spray freeze-drying process. Compared with the traditional sucrose fatty acid ester production process, the production efficiency and product quality have been improved.

[0012] Compared with the traditional powdered sucrose fatty acid ester processed by traditional mechanical grinding, this technology has the advantages of uniform particle size distribution, high sphericity, good fluidity, resistance to moisture, high wettability and solubility, and the original physical and chemical properties of the product can be effectively maintained, and the product is easy to be stored for a long time.

[0013] Due to the excellent wetting and dissolution properties of the technology, the product can be quickly dispersed and dissolved, so it can avoid the traditional powdered sucrose fatty acid ester products that are difficult to disperse and dissolve, and even the occurrence of clumping.

[0014] 4· Sucrose fatty acid ester production without mechanical crushing, to avoid dust may cause environmental pollution, product pollution, human harm and so on.

[0015 figure 1: microscopic magnification of sucrose fatty acid ester particles prepared in example 1 Picture. [0016] figure 2: microscope of sucrose fatty acid ester particles prepared by example 2 Enlarge the picture. [0017 figure 3: visualization of sucrose fatty acid ester particles prepared by example 3 Enlarge the picture with a micromirror.

Illustration with picture

[0015 图1:实施例1制备得到的蔗糖脂肪酸酯颗粒的显微镜放大图。 [0016] 图2:实施例2制备得到的蔗糖脂肪酸酯颗粒的显微镜放大图。 [0017 图3:实施例3制备得到的蔗糖脂肪酸酯颗粒的显微镜放大图。

Concrete implementation mode

[0018] Embodiments 1

1) The sucrose fatty acid ester reaction products were separated and purified by co-precipitation method to obtain refined sucrose fatty acid ester solution with 85% solid content and good flow condition at 90℃.

[0019] 2) The obtained sucrose fatty acid ester solution was pumped into the centrifugal atomizer for spray cooling. The spray cooling conditions were as follows: the atomizer rotation speed was 6000rpm, the inlet air temperature was 15℃, the outlet air temperature was 40℃, the feed temperature was 90℃, the feed speed was 50L/ h, and the cold air flow rate was 1000 cubic meters/hour.

[0020] The obtained product particle size ranges from 200 to 500m, and is a regular spherical particle, with good fluidity.

[0021] 3) The granular material is transferred to the freezer, the temperature of the freezer is -40℃, maintained for 5h, and the temperature rises to - within 1.5h

28 ° C and maintained for 30h; Then the temperature rises to -12℃ within 5h and maintains for 5h; Then heating to 23-20℃ and maintained for 7h, freezing process pressure maintained 0.01-1.0Pa, sucrose fatty acid ester particles can be prepared.

[0022] The grain size of sucrose fatty acid ester particles obtained by this method was 86% distributed between 200-500ym, and the water content was 0.5%.

[0023] Embodiment 2

1) The reaction products of sucrose fatty acid ester were separated and purified by aqueous solvent extraction method to obtain refined sucrose fatty acid ester with solid content of 85% and good flow condition at 90℃.

[0024] 2) The sucrose fatty acid ester solution is pumped into the centrifugal atomizer for cooling spray, the spray conditions are as follows: the atomizer speed is 9000rpm, the inlet air temperature is 15℃, the outlet air temperature is 40℃, the feed temperature is 90℃, the feed speed is 50L/h, and the cold air flow is 1000 cubic meters/hour.

[0025] The obtained product particle size range is between 100~400um, and it is a regular spherical particle with good fluidity.

[0026] 3) The granular material is transferred to the freezer, and the temperature of the freezer is -40℃, maintained for 5h, and then heated up to 28℃ within 1.5h and maintained for 30h; Then the temperature rises to -12℃ within 5h and is maintained for 5h; Then heating to 23~20℃ and maintained for 7h, freezing process pressure maintained 0.01~1.0Pa, sucrose fatty acid ester particles can be prepared.

[0027] The grain size of sucrose fatty acid ester particles obtained by this method was 78% distributed between 100 and 400m, and the water content was 0.5%.

[0028] Embodiment 3

1) Sucrose fatty acid ester was purified from sucrose fatty acid ester by aqueous solvent extraction. The solid content is 85% and the flow condition is good at 90℃.

[0029] 2) The sucrose fatty acid ester solution is pumped into the centrifugal atomizer for cooling spray, and the spray conditions are as follows: the speed of the atomizer is 20000rpm, the inlet air temperature is 15℃, the outlet air temperature is 40℃, the feed temperature is 90℃, and the feed speed is 30L/h. Cold air flow 1000 cubic meters/hour.

[0030] The obtained product particle size ranges from 10 to 200um, and is a regular spherical particle, with good fluidity.

[0031] 3) The granular material is transferred to the freezer, and the temperature of the freezer is -40℃, maintained for 5h, and then heated up to 28℃ within 1.5h and maintained for 30h; Then the temperature rises to -12℃ within 5h and is maintained for 5h; Then heating to 23~20℃ and maintained for 7h, the whole freezing process pressure 0.01-1.0Pa, sucrose fatty acid ester particles can be prepared.

[0032 The grain size of 91% sucrose fatty acid ester particles obtained by the method was in the range of 10~200um, and the water content was 0.8%☒

[0033] The atomizing disc diameter of the atomizer used in each embodiment of the invention is 200 mm.

[0034] In addition to the aqueous solvent extraction method and co-precipitation method, the separation and purification method of the invention can also adopt the precipitation method, or the combination of the aqueous solvent extraction method, co-precipitation method and precipitation method.

[0035] The separation and purification method of the invention can also be a molecular distillation method, a thin layer chromatography method or a liquid chromatography method.

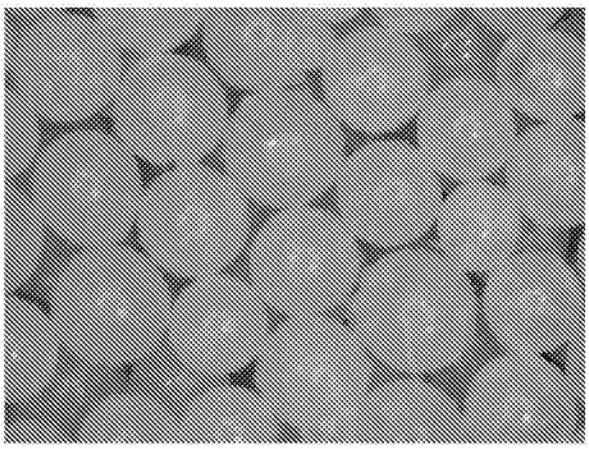

图1

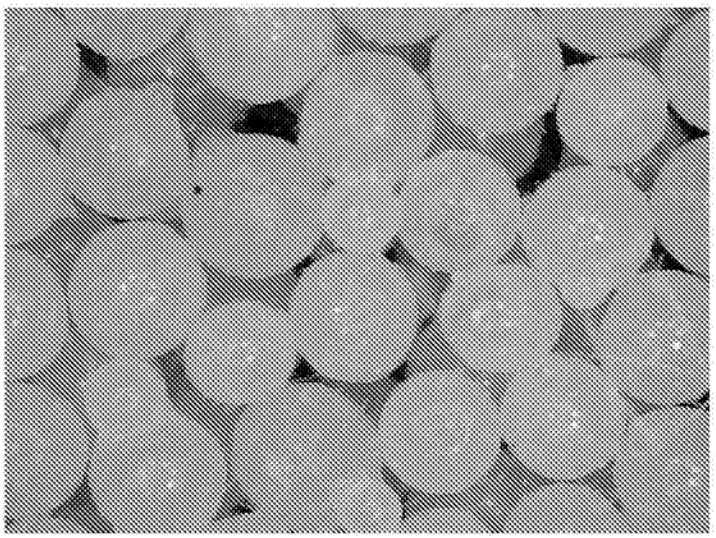

Figure 2

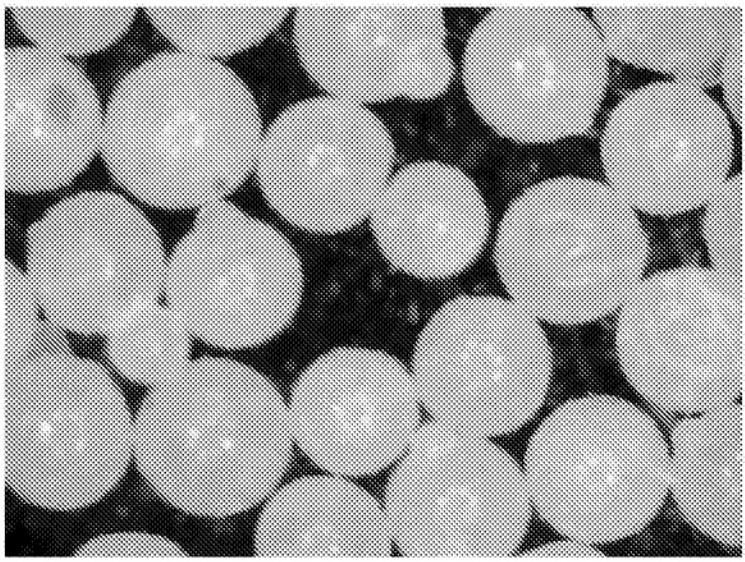

Figure 3

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(56) Compare files

CN 1995053 A, 2007. 07. 11,

US 4104464 A, 1978. 08. 01,

EP 0448996 B1, 1998. 08. 19,

CN 1654469 A, 2005. 08. 17,

Guo Bingjie, Censor

权利要求书1页 说明书4页

(54) Names

- Method of purification of sucrose fatty acid ester

(57) Abstract

The invention relates to a method for purifying sucrose fatty acid ester. The method is to dissolve the crude sucrose ester in an organic solvent that can be separated from water, obtain the crude sucrose ester solution, and add a saline solution dissolved with alkaline earth metal salt and/or alkaline earth metal oxide under agitation, so that the fatty acid soap substances in the crude sucrose ester solution can generate fatty acid alkaline earth metal salt that is insoluble in organic solvent. Then remove the generated solid fatty acid alkaline earth metal salt, and then add salt water for stirring extraction, stand to separate the lower water layer, the supernatant is cooled to 0℃-40℃ under agitation, the sucrose ester crystal precipitates in the form of precipitation, remove the liquid to take solid substances, and get the sucrose ester product after drying. The invention introduces a cooling crystallization process, removes the residual problem of unreacted raw materials

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1· A method for the purification of sucrose fatty acid ester is characterized in that it comprises the following steps:

(1) the crude sucrose ester is dissolved in an organic solvent that can be separated from water to obtain the crude sucrose ester solution;

(2) the crude sucrose ester solution is stirred at 30℃-110℃ and drops a salt solution with a concentration of 3%-20% in the mass percentage of calcium chloride or a mixture of calcium chloride and calcium oxide, stirring at 30℃-110℃ for 20-30min to generate solid fatty acid alkaline earth metal salt; The amount of calcium chloride is 3-7% of the crude quality of sucrose ester, the amount of calcium oxide is 1-2% of the crude quality of sucrose ester, and the amount of salt aqueous solution is 20%-50% of the crude quality of sucrose ester;

(3) Remove the generated solid fatty acid alkaline earth metal salt:

(4) Add 3%-20% brine with mass percentage concentration to the solution of removing solid fatty acid alkaline earth metal salt at 30℃-110℃, stir extraction at 30℃-110℃ for 10-20min, stand to separate the lower water layer, and take the superserum; The amount of salt water is 0.5-2 times the quality of sucrose ester crude product;

(5) The supernatant is cooled under stirring so that the temperature is reduced by 20-70℃ to reach the crystallization temperature of 0℃-40℃, and the sucrose ester crystal is obtained

(6) Remove the liquid to take a solid substance, after drying to obtain sucrose ester products;

Step (1) in the organic solvent is ethyl acetate, butyl ketone or n-butanol, the amount of organic solvent according to 100g sucrose ester crude add 400ml organic solvent meter;

The salt substance in the salt aqueous solution is sodium chloride or potassium chloride;

The brine described in step (4) is an aqueous solution of sodium chloride or potassium chloride.

2. A method of purification of sucrose fatty acid ester described in claim 1 is characterized by: the removal of solid fatty acid alkaline earth metal salt described in step (3) is centrifuged, precipitated or filtered; The removal of the liquid described in step (6) is by centrifugation, precipitation or filtration.

3· A method of purification of sucrose fatty acid ester described in claim 1 or 2 is characterized in that: the salt aqueous solution added in step (2) has a mass percentage concentration of 5-10%; The brine added in step (4) has a mass percentage concentration of 5-10%

- Method of purification of sucrose fatty acid ester

Technical fields

[0001] The invention relates to a method for purifying sucrose fatty acid ester.

Background technology

[0002] Sucrose fatty acid ester, also known as sucrose ester (referred to as SE), is an ester produced by esterification reaction with C9-C22 fatty acid in an appropriate system with sucrose as raw material. It has a good emulsification effect on water and oil, and the HLB value (hydrophilic lipophilic balance value) can be adjusted in a wide range. As an excellent non-ionic surfactant, it is widely used in many fields such as food, pharmaceutical and daily chemical, and has broad prospects for development.

[0003] At present, most of the industrialized methods for the synthesis of sucrose ester are transesterification, which is divided into solvent method and solven-free method. The sucrose ester products produced by these two methods contain more other substances, such as unreacted free sugars, soap bodies, fatty acids, fatty acid methyl esters or catalysts and other impurities, and the purity of the products is not high. Patent No. CN 200510006011. The invention patent of 1 discloses a method of purification of sucrose fatty acid ester, which is suitable for the purification of crude sucrose ester produced by transesterification, but the sucrose ester product obtained after purification by the method, the total ester content is only 94%, the total ester content is still low, and can not meet the user's higher requirements for product purity.

Content of the invention

[0004] The technical problem to be solved by the invention is to provide a method for purifying sucrose fatty acid ester whose total ester content in the product reaches more than 98% and improves the purity of the product. The method simplifies the production process and is more conducive to industrial production.

[0005] The technical solution to the above technical problems is: a method for purifying sucrose fatty acid ester, which comprises the following steps:

[0006] (1) the crude sucrose ester is dissolved in an organic solvent that can be separated from water to obtain the crude sucrose ester solution;

[0007 (2) The crude solution of sucrose ester is stirred at 30℃-110℃ and drops a saline solution with 3%-20% concentration of alkaline earth metal salt and/or alkaline earth metal oxide, stirring at 30℃-110℃ for 20-30min, so that the fatty acids and soap substances in the crude solution of sucrose ester generate solid fatty acids and bases that are insoluble in the organic solvent Earth metal salt; the amount of alkali earth metal salt is 3-7% of the crude quality of sucrose ester, the amount of alkali earth metal oxide is 1-2% of the crude quality of sucrose ester, and the amount of salt aqueous solution is 20%-50% of the crude quality of sucrose ester;

[0008] (3) Remove the generated solid fatty acid alkaline earth metal salt;

[000] (4) Add 3%-20% brine at 30℃-10℃ to the solution of removing solid fatty acid alkaline earth metal salt, stir at 30℃-110℃ for 10-20min extraction, stand to separate the lower water layer, and take the supernuant; The amount of salt water is 0.5-2 times the quality of sucrose ester crude product;

[0010] (5) The supernatant is cooled under agitation so that the temperature is reduced by 20-70℃ to reach the crystallization temperature of 0℃-40℃, and the sucrose ester crystal is precipitated in the form of precipitation;

[0011] (6) Remove the liquid to take a solid substance, after drying to obtain sucrose ester products.

[0012]

The organic solvents described in step (1) are ethyl acetate, methanol, ethanol, toluene, butanone, n-butanol, chloroform, or N Hexane.

[0013] The alkaline earth metal salts described in step (2) are calcium chloride or/and magnesium chloride or/and barium chloride; Said alkalis

The metal oxides are calcium oxide or/and magnesium oxide or/and oxidation; The salts in the salt solution are sodium chloride, potassium chloride, sodium sulfate, or potassium sulfate.

[0014] The brine in step (4) is an aqueous solution of sodium chloride, an aqueous solution of potassium chloride, an aqueous solution of sodium sulfate, or an aqueous solution of potassium sulfate.

[0015] The removal of the generated solid fatty acid alkaline earth metal salt described in step (3) is done by centrifugation, precipitation or filtration; The removal of the liquid described in step (6) is done by centrifugation, precipitation or filtration.

[0016] The further technical scheme of the invention is: the mass percentage concentration of the salt aqueous solution added in step (2) is 5-10%; The mass percentage concentration of the brine added in step (4) is 5-10%.

[o017] The amount of organic solvent added in step (1) is 2-7 times the mass of the crude sucrose ester.

[0018] By adopting the technical scheme, the invention has the following beneficial effects:

[0019] 1. The invention adopts a new process to remove fatty acids before they are acidified and decomposed, and solves the problem of high acid value caused by incomplete removal of fatty acids after acidification in the prior art.

[0020] 2. The invention introduces a cooling crystallization process to remove the residual problem of unreacted raw materials such as fatty acid methyl ester in the product, and further improves the content grade of the product. The total ester content of the product prepared by the invention reaches more than 98%, and the purity of the product is improved.

[0021] 3. The invention also simplifies the production process and is more conducive to industrial production

[0022] The technical characteristics of a method for purifying sucrose fatty acid ester of the present invention are further explained in combination with embodiments.

Specific embodiments

[0023] The crude sucrose ester used in embodiments 1-4 of the invention is an industrial sucrose ester product A synthesized by solventless transesterification, and the crude component is as follows:

[0024] component	content (%)
[0025] sucrose ester	55.1
[0026] Unreacted free sugars	21
[o02] soap body	15.1
[002] fatty acids	2.8
[002 fatty acid methyl ester	3.6
[0030 Catalyst and other impurities	2.4

[0031] The content of single ester in sucrose ester was 60.2%, double ester 23.7% and polyester 16.1%.

[0032 Example 1:

[003 Take 100g of the above crude sucrose ester A, add 400ml ethyl acetate (C, p), heat up to 65℃, drop and dissolve 5 grams of anhydrous calcium chloride with 5% concentration NaC1 aqueous solution 30g under agitation, stir the mixture at 65℃ for 25 minutes, filter to remove the sediment. Add 100g of 5% sodium chloride aqueous solution with mass percentage concentration at 60℃, stir at 60℃ for 15 minutes, leave to remove the lower water layer, take organic supernatant and stir, cool to 10℃, vacuum filter to remove the liquid substance, filter cake dry under pressure, obtained purified sucrose ester sample 53.2 grams. The total ester content was 98.2%, the acid value was 0.82 mgKOH/g, the free sugar was 0.47%, the ash was 0.28%, the single ester content of the sample was 56.8%.

[0034] Illustrative embodiment 2:

[0035] Take 100g of the above crude sucrose ester A, add 400ml ethyl acetate (C, p), heat up to 65℃, and drop under stirring

Add 5 grams of anhydrous calcium chloride with 5%NaC1 aqueous solution by mass percentage concentration 30g, the mixture is stirred at 65℃ for 25 minutes, filter to remove sediment. Add 100g of 5% sodium chloride aqueous solution by mass percentage concentration at 60℃, stir at 60℃ for 15 minutes, and leave to remove the lower water layer. Take the organic supernatant and stir it and cool it to 20℃, filter it under vacuum, and dry the filter cake under reduced pressure to obtain the purified sucrose ester sample of 50.5 g. The total ester content was 98.4%, acid value was 0.84 mgkOH/g, free sugar was 0.49%, ash was 0.28%. The monoester content of the sample was 59.4%

[0036] Example 3:

[0037] Take 100 grams of the above crude sucrose ester A, add 400ml ethyl acetate (C, p), heat up to 65℃, and add 5 grams of anhydrous calcium chloride in 5%NaC1 aqueous solution by mass percentage concentration 30g under agitation, stir the mixture at 65℃ for 25 minutes, filter out the sediment. Add 100g of 5% sodium chloride aqueous solution by mass percentage concentration at 60℃, stir at 60℃ for 15 minutes, and leave to remove the lower water layer. The organic supernatant was stirred and cooled to 40℃, then filtered by vacuum, and dried by filter cake under reduced pressure to obtain 42 grams of purified sucrose ester sample. The total ester content was 98.8%, the acid value was 0.82 mgKOH/g, the free sugar was 0.45% and the ash was 0.32%. The monoester content of the sample was 71.4%.

[0038] Embodiment 4:

[0039] Take 100 grams of the above crude sucrose ester A, add 400ml ethyl acetate (C, p), heat up to 65℃, and add 5 grams of anhydrous calcium chloride and calcium oxide mixture (calcium chloride: calcium oxide =4:1) under agitation w/W) mass percentage concentration 10% potassium chloride aqueous solution 50g, the mixture stirred at 65℃ for 25 minutes, filter to remove the precipitate. Add 100% concentration 10% potassium chloride aqueous solution at 65℃ 100g, stir at 65℃ for 15 minutes, stand to remove the lower water layer. The organic supernatant was stirred and cooled to 20℃, then filtered by vacuum, and dried by filter cake under reduced pressure to obtain 47 grams of purified sucrose ester sample. The total ester content was 98.4%, the acid value was 0.42 mgKOH/g, the free sugar was 0.39% and the ash content was 0.25%. The monoester content of the sample was 58.6%.

[004] The crude sucrose ester used in embodiments 5-7 of the invention is an industrial sucrose ester product B synthesized by solventless transesterification, and the crude component is as follows:

[0041] component	content (%)
[0042] Sucrose ester	68.9
[0043] Unreacted free sugars	13.3
[0044] Soap body	10.5
[0045] Fatty acids	3.1
[0046] Fatty acid methyl ester	2.2
[0047] Catalyst and other impurities	2.0

[0048] The content of single ester in sucrose ester is 22%, double ester 35% and polyester 43%.

[0049] Example 5:

[0050] Take 100 grams of the above crude sucrose ester B, add 400ml of n-butanol (C, p), heat up to 50℃, and add 5 grams of anhydrous calcium chloride and calcium oxide mixture (calcium chloride: calcium oxide =4:1) under stirring w/W) mass percentage concentration 3% potassium chloride aqueous solution 50g, the mixture stirred at 50℃ for 25 minutes, filter to remove the precipitate. At 50℃ add mass percentage concentration 3% potassium chloride aqueous solution 50g, stirring at 50℃ for 15 minutes, standing points to the lower water layer. Take the organic supernatural liquid stirred and cooled to 0℃, vacuum filtration, filter cake vacuum drying, purified sucrose ester sample 51 grams, the total ester content of 98.1%, acid value of 0.61 mgKOH/g, free sugar 0.36%, ash 0.44%, sample single ester content of 19.6%.

[0051] Embodiment 6:

[0052] Take 100 grams of the above crude sucrose ester B, add 400ml ethyl acetate (C, p), heat up to 65℃, and add 5 grams of the mixture of anhydrous magnesium chloride and calcium oxide (magnesium chloride: calcium oxide =4:1 w/w) under stirring, the mass percentage concentration of 10%

50g sodium chloride aqueous solution, mixture stirred at 65℃ for 40 minutes, filter to remove the sediment. Add 100g of 10% potassium chloride aqueous solution by mass percentage concentration at 65℃, stir at 65℃ for 25 minutes, and leave to remove the lower water layer. The organic supernatant was stirred and cooled to 10℃, then filtered by vacuum, and dried by filter cake under reduced pressure to obtain 53 grams of purified sucrose ester sample. The total ester content was 98.8%, the acid value was 0.36mgKOH/g, the free sugar was 0.39% and the ash content was 0.25%. The monoester content of the sample was 22%.

[0053] Embodiment 7:

[0054] Take 100 grams of the above crude sucrose ester B, add 400ml butanone (C, p), heat up to 55℃, and add 7 grams of anhydrous calcium chloride and calcium oxide mixture (calcium chloride: calcium oxide =4:1) under stirring w/W) mass percentage concentration of 8% sodium chloride aqueous solution 50g, the mixture at 55℃ stirred 25 minutes, filter to remove the sediment. Add 100g mass percentage concentration 8% sodium chloride aqueous solution at 55℃, stir for 20 minutes at 55℃, and leave to remove the lower water layer. The organic supernatant was stirred and cooled to 10℃, then filtered by vacuum, and dried by filter cake under reduced pressure to obtain 49 grams of purified sucrose ester sample. The total ester content was 98.8%, acid value was 0.41 mgKOH/g, free sugar was 0.21%, ash was 0.15%. The monoester content of the sample was 20.4%.

[0055] The organic solvents added in each embodiment of the invention may be ethyl acetate, methanol, ethanol, toluene, butanone, n-butanol, chloroform or n-hexane; The alkali earth metal salts added may be calcium chloride or/and magnesium chloride or/and barium chloride (barium salt is toxic, so calcium chloride or/and magnesium chloride are preferred); The added alkaline earth metal oxides are calcium oxide or/and magnesium oxide or/and barium oxide (the oxides are toxic, so it is preferred to calcium oxide or/and magnesium oxide); The salts in the brine may be sodium chloride, potassium chloride, sodium sulfate or potassium sulfate.