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(54) Name of the invention

A purification and separation method of sucrose fatty acid ester

(57) Summary

The invention discloses a purification and separation method of sucrose fatty acid ester,

including the following steps:ADissolve crude sucrose fatty acid esters and disperse them in organic solventsAIn the middle, the crude solution of sucrose fatty acid ester is obtained after filtration and recovery of sucrose;B, sucrose fatty acid ester crude product solution in25One80 ℃After stirring, add alkaline earth metal salts for a complex decomposition reaction, and then5One80 ℃The filtrate is obtained by solid-liquid separation.AAnd solidB;Centigrade, filtrateAWash and distillate to recover solvents and dry to obtain sucroseFatty ester productsA;Dimension, solidBAdd organic solventsBAt50One80 ℃Extraction, the extraction liquid is washed, distilled to recover the solvent, and dried to obtain sucrose fatty acid ester products.B. The invention has a simple process, less use of organic solvents, few side reactions, high product content and recovery rate, and the separation of sucrose and double esters at the same time.=To products with different sucrose monoester content, and in crude sucrose esters ㎝ The sucrose is recyclable and has a small amount of waste and is easy to treat.0Cun Point.

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1 ·A purification and separation method of sucrose fatty acid ester, including the following steps:

ADissolve crude sucrose fatty acid esters and disperse them in organic solventsAIn the middle, the crude solution of sucrose fatty acid ester is obtained after filtering and recovering sucrose; the crude product of sucrose fatty acid ester mainly contains sucrose fatty acid ester, sucrose and fatty acid potassium salt or sodium salt; the organic solventASelected from one or any of the following combinations: ethyl acetate, butyl ketone, n-butanol;

B, sucrose fatty acid ester crude product solution in25One80 ℃After stirring, add alkaline earth metal salts for a complex decomposition reaction, and then5One80 ℃The filtrate is obtained by solid-liquid separation.AAnd solidB; The alkaline earth metal salts are selected from one or any combination of the following: magnesium sulfate, calcium chloride and barium chloride;

c、 FiltrateAWashed, distilled and recycled solvents and dried sucrose fatty acid ester productsA;

d、 SolidBAdd organic solventsBAt50One80 ℃Extraction, the extraction liquid is washed, distilled to recover the solvent, and dried to obtain sucrose fatty acid ester products.B: The organic solvent mentionedBSelected from one or any combination of the following: ethyl acetate, butyl ketone, n-butyl

2 ·According to the claim1The purification and separation method of sucrose fatty acid ester is characterized by: the stepsAMedium, organic solventAThe volume and dosage is calculated as the quality of the crude sucrose fatty acid ester.2,5mL/g.

3 ·According to the claim1Someone2The purification and separation method of sucrose fatty acid ester is characterized by the fact that the amount of alkali earth metal salt is required to fully react with the fatty acid salt in the crude sucrose fatty acid ester.1One1 · 05Double。

4 ·According to the claim1Someone2The purification and separation method of sucrose fatty acid ester is characterized by: the compound decomposition reaction time isTen,100Minutes.

5 ·According to the claim4The purification and separation method of sucrose fatty acid esters is characterized by: steps (B), the solid-liquid separation is in30One65 ℃Go on.

6 ·According to the claim1The purification and separation method of sucrose fatty acid ester is characterized by: the stepsDimensionMedium, organic solventBThe volume and dosage is calculated by the quality of the crude sucrose ester.2,5mL/g.

7 ·According to the claim1The purification and separation method of the sucrose fatty acid ester is characterized by the following steps:

a、 Dissolve and disperse crude sucrose fatty acid esters in organic solventsAIn the middle, the crude solution of sucrose fatty acid ester is obtained after filtering and recovering sucrose; the crude product of sucrose fatty acid ester mainly contains sucrose fatty acid esters, sucrose and fatty acid salts; the organic solventASelected from one or any combination of the following: ethyl acetate, butyl ketone, n-butanol; organic solventAThe volume and dosage is calculated as the quality of the crude sucrose fatty acid ester.2,5mL/g;

b、 Sucrose fatty acid ester crude solution in25One80 ℃After stirring, add alkaline earth metal salt for compound decomposition reaction. The amount of alkaline earth metal salt is the amount required to fully react with the fatty acid salts in the crude sucrose fatty acid ester.1One1 · 05Double, the reaction time of complex decomposition isTen,100Minutes; and then in30One65 ℃The filtrate is obtained by solid-liquid separation.AAnd solidB:Centigrade, filtrateAWashed, distilled and recycled solvents and dried sucrose fatty acid ester productsA:

Dimension, solidBAdd organic solventsBAt50One80 ℃Extraction, the extraction liquid is washed, distilled to recover the solvent, and dried to obtain sucrose fatty acid ester products.B; the organic solvent mentionedBSelected from one or any combination of the following: ethyl acetate, butyl ketone, n-butyl alcohol, organic solventBThe volume and dosage is calculated by the quality of the crude sucrose ester.2,5mL/g.

A purification and separation method of sucrose fatty acid ester

Technical field

[0001]The invention relates to a purification and separation method of sucrose fatty acid ester.

Background technology

[0002]At present, the method of industrial production of sucrose fatty acid esters (hereinafter referred to as sucrose esters) adopts the ester exchange method, and the ester exchange method is divided into solvent method and solvent-free method. However, the crude sucrose esters obtained by these two methods contain different contents of sucrose monoesters, sucrose diesters, sucrose polyesters and unreacted sucrose. In order to obtain products with high sucrose ester content, impurities such as fatty acid methyl (or ethyl) ester and fatty acid potassium (or sodium) must be purified: due to the limitations of the synthesis process, it can only be synthesized.20One55Products with % sucrose monoester content, and products with higher or lower sucrose monoester content must be separated from sucrose monoesters. The existing technology is patented by Tetsuo Ishisu and others in Japan (the concession communiqué)51One14487) describes a purification and separation method of sucrose fatty acid ester, which is100The crude sucrose ester by weight is dissolved in1000In the weight of butyl ketone, then add acid to convert fatty acid soap into fatty acid, in60 ℃Join downTenWeight sodium chloride to produce precipitation and separate precipitation.AAnd filtrateB; It will precipitate again.ABy means of100Weight of butyl ketone and100The weight is dissolved in water, the water layer is removed and washed, and the butyl ketone layer is distilled and dried to obtain sucrose ester with high sucrose monoester content.A: In addition to the filtrateBAdd inTenWeight of sodium chloride and cooled to5 ℃To produce precipitation, separate the precipitation, and then follow the precipitation.ASucrose ester with low sucrose monoester contentBThis method not only completes the purification of sucrose esters, but also completes the separation of sucrose monoesters and diesters. However, the method has the following problems:A·The process is complex and unfavorable to industrial production;B ·When acidified, the sucrose ester itself is easily hydrolyzed under acidic and alkaline conditions, and the hydrolysis is particularly serious when the temperature is high, resulting in a low product recovery rate;C ·Large amount of solvent use;D ·The sucrose in crude sucrose esters cannot be recycled and used. On the one hand, it increases the cost, and on the other hand, theChemical oxygen demandSignificantly improved•,E ·Due to the addition of a

large amount of sodium chloride and the production of a large amount of high-salt wastewater, this also leads to environmental protection.

Invention content

[0003]The technical problem to be solved by the invention is that it can overcome the defects of the above-mentioned existing technology and provide a purification and separation method of sucrose fatty acid ester. It has a simple process and is convenient for industrial production, few side reactions, high product content and recovery rate, and can be different from the separation of sucrose monoesters at the same time. Products with sucrose monoester content, and the sucrose in crude sucrose ester can be recycled and has the advantages of small waste and easy to handle.

[0004]In order to solve the above technical problems, the invention adopts the following technical solutions:

[0005]A purification and separation method of sucrose fatty acid ester, including the following steps:

[0006] aDissolve and disperse sucrose ester crude products in organic solventsAIn China, the crude solution of sucrose ester is obtained after filtering and recovering sucrose; the crude sucrose ester mainly contains sucrose ester, sucrose and fatty acid potassium salt or sodium salt; the organic solventASelected from one or any of the following combinations: ethyl acetate, butyl ketone, n-butanol;

[0007] b, sucrose ester crude solution in25One80 ℃After stirring, add alkaline earth metal salts for a complex decomposition reaction, and then5One

80 ℃The filtrate is obtained by solid-liquid separation.AAnd solidB;

[0008] c, filtrateAWashed, distilled, recycled solvents, dried sucrose ester productsA;

[0009] d, solidBAdd organic solventsBAt50One80 ℃Extraction, the extraction liquid is washed, distilled to recover the solvent and dried.

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Sucrose ester productsB: The organic solvent mentionedBSelected from one or any combination of the following: ethyl acetate, butyl ketone, butyl alcohol.[0010]The crude sucrose ester used in the present invention can be the crude sucrose ester synthesized from the water solvent method or solvent-free method, especially suitable for the crude sucrose ester with high content of fatty acid salts (fatty acid potassium or sodium).

[0011]The steps describedAMedium, organic solventAThe volume and dosage is calculated by the quality of the crude sucrose ester.2,5mL/g.

[0012]The steps describedBIn the middle, alkaline earth metal salt is added to the crude solution of sucrose ester, which is decomposed with the fatty acid salt (potassium or sodium fatty acid) in the solution to produce insoluble fatty acid alkaline earth metal salt and another alkali metal salt (potassium or sodium salt). They form coprecipitate with sucrose ester, and sucrose monoester is easier to form than sucrose bisester. Co-precipitates, so sucrose monoesters are concentrated in co-precipitates to achieve the purpose of separating sucrose monoesters and sucrose biesters. Further, the alkaline earth metal salt can be selected from one or any combination of the following: magnesium sulfate, calcium chloride, and barium chloride. The amount of alkaline earth metal salt is required to fully react with the fatty acid salt in the crude sucrose ester.1One1 · 05Double. The complex decomposition reaction time is preferred asTen,100Minutes. The solid-liquid separation is preferred in30One65 ℃Go on.

[0013]The steps describedDimensionMedium, organic solventBThe volume and dosage is calculated by the quality of the crude sucrose ester.2,5mL/g.

[0014]The purification and separation method of sucrose fatty acid esters specifically recommended by the invention shall be carried out in the following steps:

[0015] aDissolve crude sucrose fatty acid esters and disperse them in organic solventsAIn the middle, the crude solution of sucrose fatty acid ester is obtained after filtering and recovering sucrose: the crude product of sucrose fatty acid ester mainly contains sucrose fatty acid esters, sucrose and fatty acid salts; the organic solventASelected from one or any combination of the following: ethyl acetate, butyl ketone, n-butanol: organic solventAThe volume and dosage is calculated as the quality of the crude sucrose fatty acid ester.2,5mL/g;

[0016] b, sucrose fatty acid ester crude product solution in25One80 ℃After stirring, alkaline earth metal salts are added for repeated decomposition. The alkaline earth metal salts are selected from one or any of the following combinations: magnesium sulfate, calcium chloride, and barium chloride. The amount of alkaline earth metal salt is the amount required to completely react with the fatty acid salts in the crude sucrose fatty acid ester.1One1 · 05Double, the reaction time of complex decomposition is Ten,100Minutes; and then in30One65 ℃The filtrate is obtained by solid-liquid separation.AAnd solidB;

[0017] c, filtrateAWashed, distilled and recycled solvents and dried sucrose fatty acid ester productsA;

[0018] d, solidBAdd organic solventsBAt50One80 ℃Extraction, the extraction liquid is washed, distilled to recover the solvent, and dried to obtain sucrose fatty acid ester products.B: The organic solvent mentionedBSelected from one or any combination of the following: ethyl acetate, butyl ketone, n-butyl alcohol, organic solventBThe volume and dosage is calculated by the quality of the crude sucrose ester.2,5mL/g.

[0019]Compared with the existing technology, the invention has a simple process, less use of organic solvents, less side reactions, wide-mouth content and high recovery rate, and completes the separation of sucrose and double esters at the same time to obtain different sucrose monoester content. Moreover, the sucrose in the crude sucrose ester is recyclable and the three waste is small and easy to treat. Point.

Specific implementation methods

[0020]The following embodiments are further elaborated on the invention in combination with the implementation examples. The following embodiments are a detailed explanation of the invention, and the invention is not limited to the following embodiments.

[0021]The percentage of the components of crude sucrose ester in the embodiment of the invention is the percentage of quality.

[0022]Example 1**:**

[0023]Grinded sucrose ester crude product (synthesized by solvent-free method, composition: sucrose ester47 · 1%, potassium stearate25 · 9%, sucrose24 · 8%, other2 · 2%)100gLoad in500mLIn the three-neck flask, add250mLEthyl acetate, stir to heat up to70 ℃, thermal insulation and dissolution30After a few minutes, filter out the undissolved sucrose; then60 ℃Stir and add it to the filtrate.15gMagnesium sulfate aqueous solution (containing magnesium sulfate)5 · Og), thermal insulation reaction1In hours, filter and separate to obtain the filtrate.AAnd solidB; FiltrateAWash with water (60mLx3), after the upper organic layer is distilled to recover ethyl acetate, sucrose ester is obtained after vacuum drying.A18 · 5g(Sucrose ester content94 · 2%, of which sucrose monoesters contain/

Measure28 · 5%): solidBAdd in300mLEthyl acetate60 ℃Extraction, the extraction liquid is washed with water (80mLx3), after the upper organic layer is distilled to recover ethyl acetate, sucrose ester is obtained after vacuum drying.B27 · 8g(Sucrose ester content96 · 1%, of which the content of sucrose monoester58 · 2%).

[0024]Example 2**:**

[0025]Grinded sucrose ester crude product (synthesized by solvent-free method, composition: sucrose ester52 · 9%, sodium stearate21 · 0%, sucrose

23 · 3%, other2 · 8%)100gLoad in500mLIn the three-neck flask, add260mLButyl ketone, stir and heat up to70 ℃, thermal insulation and dissolution

30After a few minutes, filter out the undissolved sucrose; then55 ℃Stir and add it to the filtrate.10gCalcium chloride aqueous solution (calcium chloride)4 · Og), thermal insulation reaction1In hours, filter and separate to obtain the filtrate.AAnd solidB; FiltrateAWash with water (60mLx3), after the upper organic layer distillation is recovered,

the sucrose ester is obtained by vacuum drying.A19 · 6g(Sucrose ester content93 · 8%, of which the content of sucrose monoester18 · 8%); solidBAdd in300mLButyl ketone70 ℃Extraction, the extraction liquid is washed with water (80mLx3), after the upper organic layer distillation is recovered, the sucrose ester is obtained by vacuum drying.B32 · 7g(Sucrose ester content96 · 5%, of which the content of sucrose monoester55 · 3%).

[0026]Example 2**:**

[0027]Grinded sucrose ester coarse product (synthesized by water solvent method, composition: sucrose ester49 · 8%, potassium stearate20 · 1%, sucrose27 · 5%, other2 · 6% month00gLoad in500mLIn the three-neck flask, add250mLN-butanol, stir and heat up to70 ℃, thermal insulation and dissolution30After a few minutes, filter out the undissolved sucrose; then50 ℃Stir and add it to the filtrate.28gAqueous solution of barium chloride (banium chloride)6 · 7g), thermal insulation reaction1In hours, filter and separate to obtain the filtrate.AAnd solidB; FiltrateAWash with water (80mLx3), after the upper layer of organic distillation to recover n-butanol, vacuum drying to obtain sucrose esters.A19 · l g(Sucrose ester content95 · 3%, of which the content of sucrose monoester21 · 5%); solidBAdd in320mLN-butanol in75 ℃Extract and wash the extract with water.000mLX3), after the upper layer of organic distillation to recover n-butanol, vacuum drying to obtain sucrose esters.B29 · 4g(Sucrose ester content96 · 4%, of which the content of sucrose monoester72 · 4%).

[0028]Example 4:

[0029]Grinded sucrose ester coarse product (synthesized by water solvent method, composition: sucrose ester48 · 6%, potassium palmitate20 · 4%, sucrose28 · 9%, other2 · 1%)100gLoad in500mLIn the three-neck flask, add250mLEthyl acetate, stir to heat up to70 ℃, thermal insulation and dissolution30After a few minutes, filter out the undissolved sucrose; then52 ℃Stir and add it to the filtrate.9 · 5gCalcium chloride aqueous solution (calcium chloride)3 · 8g), thermal insulation reaction1In hours, filter and separate to obtain the filtrate.AAnd solidB; FiltrateAWash with water (60mLx3), after the upper organic layer is distilled to recover ethyl acetate, sucrose ester is obtained after vacuum drying.A17 · 8g(Sucrose ester content94 · 6%, of which the content of sucrose monoester17 · 8%); solidBAdd in350mLEthyl acetate70 ℃Extraction, the extraction liquid is washed with water (80mLx3), after the upper organic layer is distilled to recover ethyl acetate, sucrose ester is obtained after vacuum drying.B29 · 8g(Sucrose ester content96 · 8%, of which sucrose monoester contains the amount75 · 2%).

[0030]Example 5:

[0031]Grinded sucrose ester crude product (synthesized by solvent-free method, composition: sucrose ester62 · 9%, potassium stearate16 · 0%, sucrose

18 · 5%, other2 · 6% month00gLoad in500mLIn the three-neck flask, add250mLButyl ketone, stir and heat up to70 ℃, thermal insulation and dissolution

30After a few minutes, filter out the undissolved sucrose; then45 ℃Stir and add it to the filtrate.9 · 3gMagnesium sulfate aqueous solution (magnesium sulfate)3 · l g), thermal insulation reaction1In hours, filter and separate to obtain the filtrate.AAnd solidB; FiltrateAWash with water (60mLx3), after the upper organic layer distillation is recovered, the sucrose ester is obtained by vacuum drying.A38 · 7g(Sucrose ester content94 · 8%, of which the content of sucrose monoester20 · 2%); solidBAdd in300mLButyl ketone65 ℃Extraction, the extraction liquid is washed with water (80mLx3), after the upper organic layer distillation is recovered, the sucrose ester is obtained by vacuum drying.B26 · 7g(Sucrose ester content98 · 2%, of which the content of sucrose monoester54 · 3%).

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Claims1Page instruction manual4Page

(54) Name of the invention

A preparation method of sucrose benzoate

(57) Summary

The invention discloses a preparation method of sucrose parabensate, which first reacts sucrose with sodium hydroxide in water to obtain an aqueous solution of sucritol sodium. The aqueous solution of sodium

sucritol is added to benzoyl chloride for the reaction. As the reaction proceeds, a certain amount of aqueous solution of toluene and sodium hydroxide is added to continue the reaction until it is complete. By adjusting the ratio of sucrose, benzoyl chloride and sodium hydroxide, the average degree of substitution can be obtained in3,6Sucrose bens with a specific degree of substitution. Compared with the existing method, the invention is stable because it is made of a highly active sucrose sodium intermediate.3,6Sucrose parabens with a specific degree of substitution betweenThe uniform substitution degree and the content of each component fluctuate less. And the operation is simple, the process stability is high, and the color of the product is low and stable, which is suitable for industrial production.

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Claims

1.An average degree of substitution is3,6The preparation method of sucrose and benzoate is characterized by the method: sucrose and sodium hydroxide.AReaction in water to obtain an aqueous solution of sodium sucrose; sucrose and sodium hydroxideAThe proportion of the amount of substances is1:1,3; Under continuous stirring, the aqueous solution of the sucritol sodium is added to the benzoyl chloride for the reaction, and the temperature of the reaction liquid is controlled.0,15 ℃During the reaction process, the reaction system becomes thicker, and when the reaction system is so thick that it is difficult to stir, toluene is added to the reaction system to reduce the viscosity of the reaction system. After the aqueous solution of sucrose sodium is added, sodium hydroxide is added.BThe temperature of the aqueous solution to control the

reaction liquid is0,15 ℃,pHValue8,1 1After adding it, it will heat up to20,25 ℃, thermal insulation reaction1,3hThe reaction is complete, and then heat up to65,70 ℃, remove the water layer, wash the organic layer with a small amount of water to be neutral, and then distilled toluene, and the remainder is sucrose benzoate; the proportion of the amount of sucrose, benzoyl chloride and total sodium hydroxide is1:3,

6:3 ～ 6. 2。

2 ·According to the claim1The method is characterized by the use of sucrose and sodium hydroxide.AWhen reacting in water, the amount of water is the quality of sucrose.0 · 5,3 倍。

3 ·According to the claim1The method is characterized by the fact that the total amount of toluene in the reaction system is sucrose mass.3,8Double。

4·According to the claim1The method is characterized by the fact that the organic layer is washed with water, and the quality and amount of the water is the quality of sucrose.1. 5,2Double.

5 ·According to the claim1The method is characterized by the ratio of the amount of sucrose and benzoyl chloride.1:3. 5,5 · 8.

6 ·According to the claim1The method is characterized by the ratio of the amount of sucrose and total sodium hydroxide.1:3 · 7,6. 1。

A preparation method of sucrose benzoate

(一) Technical field

[0001]The invention belongs to the chemical field, specifically a preparation method of sucrose benzoate.

(二) Scenery technology

[0002]As a synthetic chemical raw material, coating and cosmetic raw material, and used as a high-performance plastic modified additive, it is used as a high-performance liquid crystal display film optical material (which can give the film specific optical performance), holographic photosensitive material, electronic image recording original, and ribbon for heat transfer printing and magnetic heat transfer printing. Electrostatic sprayed toner and thermal printing ink, etc. In different applications, there are corresponding requirements for the average replacement degree and component content of sucrose parabens to achieve better use effect.

[0003]American patentUS3198784It is reported that the ratio of sucrose to benzoyl chloride is1:6,Ten, the average replacement degree of preparation is6,8The method of sucrose parabens: the esterification reaction of sucrose is carried out in solvents such as benzene, toluene, xylene, ethylbenzene, chlorobenzene, chlorbenzyl, chloromethane, chloroform and carbon tetrachloride. After the reaction is completed, the average replacement degree is obtained by refining operations such as water separation, washing, and recycling solvents.6,8The sucrose parabens. Japanese patent52One95625It is reported that the average degree of substitution prepared in organic solvents with a certain solubility in water, such as acetone, butyl ketone, dioxane, methyl acetate and tert-butyl alcohol, is1. 51,7 · 76The method of sucrose parabens.

[0004]First of all, the method described in the U.S. patent can only produce an average degree of substitution in6,8The average degree of substitution cannot be obtained.3,6Sucrose parabensate, secondly, low utilization rate of sucrose and benzoyl chloride, low product yield, and wastewaterChemical oxygen demandHigher. Although the Japanese patent synthesizes the average replacement degree,1. 51,7 · 76However, the amount of water and organic solvent of this method is large, and the solubility of the organic solvent used is large and the boiling point is low, so there is a large amount of wastewater and wastewater per unit product.Chemical oxygen demandHigh, large solvent consumption and high cost.

(三) Invention content

[0005]The technical problem to be solved by the invention is that the existence of existing technology cannot be solved with an average degree of substitution.3,6The problems of sucrose parabens, low yield and high production cost may be able to produce an average

degree of substitution.3,6However, there is a large amount of wastewater per unit product and wastewater.Chemical oxygen demandHigh, large solvent consumption and high cost. The purpose of the invention is to provide a method that can not only produce the average substitution degree in a simple, effective and stable way.3,6Sucrose benzoate with a specific degree of substitution, and high yield, low solvent single consumption, low solvent residue rate in wastewater, low amount of wastewater per unit product and wastewaterChemical oxygen demandLow, low impact on the environment, suitable for industrial production.

[0006]The technical scheme adopted by the invention is as follows:

[0007]A preparation method of sucrose parabens, the method is: sucrose and sodium hydroxideAReaction in water to obtain an aqueous solution of sucritol sodium; under continuous stirring, the aqueous solution of sodium sucritol is added to benzoyl chloride for reaction, and the temperature of the reaction liquid is controlled.0,15 ℃During the reaction process, the reaction system becomes thicker, and when the reaction system is so thick that it is difficult to stir, toluene is added to the reaction system to reduce the viscosity of the reaction system. After the aqueous solution of sucrose sodium is added, sodium hydroxide is added.BThe temperature of the aqueous solution to control the reaction liquid is0,15 ℃,pHValue8,1 1After adding it, it will heat up to20,25 ℃, thermal insulation reaction1,3hComplete reaction (optimal insulation reaction)2h), then warm up to65,70 ℃, remove the aquifer, wash the organic layer with a small amount of water to neutral, and then distillate to recycle toluene, the residue is sucrose parabens; the substances of sucrose, benzoyl chloride, and total sodium hydroxide.

The proportion of quantity is1:3,6:3,6 · 2. The average substitution degree of sucrose parabens is3,6.

[0008]In the method of the invention, the sodium hydroxideAAnd sodium hydroxideBIt is sodium hydroxide added in different steps, using sodium hydroxide.AAnd sodium hydroxideBDistinguish it, sodium hydroxideAAnd sodium hydroxideBThe total amount is the total amount of sodium hydroxide. Preferred, sucrose and sodium hydroxideAThe proportion of the amount of substances is1:1,3, more preferred as1:2,2 · 6.

[0009]The ratio of the amount of sucrose and benzoyl chloride is preferred.1:3 · 5,5 · 8.

[0010]The ratio of the amount of sucrose and total sodium hydroxide is preferred.1:3 · 7,6 · 1.

[0011]The invention describes the use of sucrose and sodium hydroxide.AWhen reacting in water, the amount of water is usually the quality of sucrose.0 · 5,3, times

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[0012]The organic layer is washed to neutral with a small amount of water, and the amount of water is usually of sucrose quality.1 . 5,2Double,可将Add water many times for washing, which is easy to wash effectively. And the washed water can also be used for the next batch of synthetic water or for washing.

[0013]In the reaction system of the present invention, with the continuous progress of the reaction, the viscosity of the reaction system continues to increase, which increases the difficulty of stirring the mixed reaction raw materials. Therefore, it is necessary to add toluene to reduce the viscosity of the reaction system, facilitate the full stirring of mixed reaction raw materials, and prevent the uneven dispersion of raw materials from causing excessive substitution. Monitor the reaction system during the reaction process, and according to the needs of the reaction system,

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oluene is added when the reaction system is so thick that it is difficult to stir. Usually, the amount of toluene added to the reaction system is sucrose quality.38Double.

[0014]The reaction principle of the invention is: first, the sucrose is reacted with a certain amount of sodium hydroxide in water to obtain the aqueous solution of sucritol sodium, which improves the sucrose reaction activity. When the aqueous solution of sucritol sodium is added to the benzoyl chloride reaction, benzoyl chloride plays the dual role of reaction agent and solvent, that is, sucritol sodium and benzoyl chloride quickly. The reaction produces the corresponding sucrose benzoate. When the aqueous solution of sucrose sodium is added, all sucrose is reacted to form.1Someone2Substituted sucrose parabens, as the reaction progresses, an appropriate amount of toluene is added to dissolve the reaction.2One3Replace sucrose parabens and reduce the viscosity of the system to facilitate2One3The substituted sucrose parabens further reacts with benzoyl chloride to form the target product. Therefore, the average replacement degree of preparation can be achieved by adjusting the ratio of sucrose and benzoyl chloride.3,6The purpose of sucrose parabens with a specific degree of substitution.

[0015]Compared with the existing method, the invention has the following significant advantages:1Because a highly active sucritol sodium intermediate is made first, it can be prepared stably by adjusting the ratio of sucrose and benzoyl chloride.3,6There is a specific degree of substitution of sucrose bens, and the average degree of substitution and the content of each component fluctuate less.2The operation is simple, the process stability is high, and the product is low and stable, which is suitable for industrial production.3Due to the improvement of reaction activity and uniformity, the utilization rate of sucrose and benzoyl chloride is improved.4The solvent consumption is low and the amount of wastewater is low, which reduces the production cost and reduces the impact on the environment.

(IV) Specific implementation methods

[0016]The invention is further explained in combination with embodiments below, but the scope of protection of the invention is not limited to this. Any simple modification, equivalent change and modification of the embodiment based on the technical essence of the invention falls within the scope of protection of the invention.

[001 7]Example 1

[0018]At250m1Add sucrose to the conical bottle.40 · Og 0 16 · 8mmo l), water60m1,48% sodium hydroxide aqueous solution20g, Yu35,40 ℃Stir to dissolve the light yellow transparent sucritol sodium aqueous solution, and transfer it to the constant pressure droplet funnel for later use. It is equipped with ice baths, agitators, thermometers and the aforementioned constant pressure droplet funnel.500m1Add benzoyl chloride to the four-neck bottle.57 · 9g(412 · 1" (1)Control the material temperature5,10 ℃Add the above aqueous solution of sucritol sodium. As the reaction progresses, the material gradually thickens and slowly adds toluene.180m1, to reduce viscosity; sucritol sodium aqueous solution about30Add the drops in a minute, and then add them to the constant pressure droplet funnel.48% sodium hydroxide aqueous solution16gAdd it to the reaction system.48% sodium hydroxide aqueous solution, while controlling the material temperature5,10 ℃,PI-18,1 1, Jiabi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time.20m1, wash4Secondary (washing water is used for the next batch of synthesis or washing). After the organic layer is distilled to recover the toluene, it is poured into a stainless steel tray while it is hot. After cooling, the transparent glass-like solid sucrose parabens are obtained.79 · l g, measured the average degree of substitution3 · 5, the utilization rate of sucrose95 · 8%, acyl chloride utilization95 · 1%.

[0019]Example 2

[0020]At250m1Add sucrose to the conical bottle.40 · Og(116 · 8" (1), water50ml,48% sodium hydroxide aqueous solution25g, Yu35,40 ℃Stir to dissolve the light yellow transparent sucritol sodium aqueous solution, and transfer it to the constant pressure droplet funnel for later use. It is equipped with ice baths, agitators, thermometers and the aforementioned constant pressure droplet funnel.500m1Add benzoyl chloride to the four-neck bottle.74 · 4g(529 · 5" (1)Control the material temperatureTen,15 ℃Add the above aqueous solution of sucritol sodium. As the reaction progresses, the material gradually thickens and slowly adds toluene.200mLTo reduce viscosity; sucrate sodium aqueous solution

is about40Add the drops in a minute, and then add them to the constant pressure droplet funnel.48% sodium hydroxide aqueous solution21. l g,Add to the reaction system48% sodium hydroxide aqueous solution, while controlling the material temperatureTen,15 ℃,PH8,1 1, Jiabi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time.20m1, wash3Secondary (washing water is used for the next batch of synthesis or washing). After the organic layer is distilled to recover the toluene, it is poured into a stainless steel tray while it is hot. After cooling, the transparent glass-like solid sucrose parabens are obtained.91. l g, measured the average degree of substitution4 · 5, the utilization rate of sucrose96 · 2%, acyl chloride utilization95 · 5%.

[0021]Example 3 of implementation

[0022]At250m1Add sucrose to the conical bottle.32 · Og(93 · 5mmol), water50m1,48% sodium hydroxide aqueous solution22 · 5g, Yu35,40 ℃Stir to dissolve the light yellow transparent sucritol sodium aqueous solution, and transfer it to the constant pressure droplet funnel for later use. It is equipped with ice baths, agitators, thermometers and the aforementioned constant pressure droplet funnel.500m1Add benzoyl chloride to the four-neck bottle.76 · l g(542 · Immol)Control the material temperature0,5 ℃Add the above aqueous solution of sucritol sodium. As the reaction progresses, the material gradually thickens and slowly adds toluene.200m1, to reduce viscosity; sucritol sodium aqueous solution about30Add the drops in a minute, and then add them to the constant pressure droplet funnel.48% sodium hydroxide aqueous solution25gAdd it to the reaction system.48% sodium hydroxide aqueous solution, while controlling the material temperature0,5 ℃,PH8,1 1, Jiabi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time.20ml,Rinsing3Secondary (washing water is used for the next batch of synthesis or washing). After the organic layer is distilled to recover the toluene, it is poured into a stainless steel tray while it is hot. After cooling, the transparent glass-like solid sucrose parabens are obtained.85 · Og,Measured average replacement degree5 · 7, the utilization rate of sucrose97 · 2%, benzoyl chloride utilization rate95 · 6%.

[0023]Implementation Example 4

[0024]At500LAdd sucrose to the reactor.128Kg(373 · 9mol), water200Kg,48% sodium hydroxide aqueous solution

64Kg, stir and heat up to35,40 ℃, dissolve the light yellow transparent sucritol sodium aqueous solution and transferred to a high-level metering tank for later use. At1000LAdd benzoyl chloride to the reactor.278 · 0Kg 0978 · 9mol), turn on the frozen salt water and control the material temperature.5,10 ℃Stir and add the

above sucrose alcohol sodium aqueous solution to control the drop acceleration to keep the material temperature at5,10 ℃As the reaction progresses, the system gradually thickens and slowly adds toluene.400Kg, to reduce viscosity; sucritol sodium aqueous solution about2One3The hourly drip is finished; then slowly drip through the high-level measuring tank.48% sodium hydroxide aqueous solution107 · 2Kg,Control the material temperature at the same time.5,10 ℃,PH8,11, add to slowly heat up to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, divide the water layer, and then add toluene.200KgUse water every time.50KgWash it with water and need to wash it.4Second (washing water is used for the synthesis of the next batch of products) andPHIt is neutral. The organic layer is transferred to500LThe reactor distillates and recycles toluene, and then cools it in a stainless steel tray while it is hot to obtain transparent glass-like solid sucrose benzoate.32L 2Kg, measured the average degree of substitution5 · 3, the utilization rate of sucrose96 · 1%, acyl chloride utilization96 · 3%.

[0025]Example 5 of implementation

[0026]At500LAdd sucrose to the reactor.128Kg(373 · 9mol)Implement example 4 recycled washing water200Kg,48% sodium hydroxide aqueous solution64Kg, stir and heat up to35,40 ℃, dissolve the light yellow transparent sucritol sodium aqueous solution and transferred to a high-level metering tank for later use. At1000LAdd benzoyl chloride to the reactor.277 · 5Kg0975 · Omi),Open the frozen brine to control the material temperature.5,10 ℃Stir and add the above sucrose alcohol sodium aqueous solution to control the drop acceleration to keep the material temperature at5,10 ℃As the reaction progresses, the system gradually thickens and slowly adds toluene.400Kg, to reduce viscosity; sucritol sodium aqueous solution about2One3The hourly drip is finished; then slowly drip through the high-level measuring tank.48% sodium hydroxide aqueous solution106 · 5Kg,Control the material temperature at the same time.5,10 ℃,PH8,1 1, add to slowly heat up to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, divide the water layer, and then add toluene.200KgUse water every time.50KgWash it with water and need to wash it.4Second (washing water is used for the synthesis of the next batch of products) andPHIt is neutral. The organic layer is transferred to500LThe reactor distillates and recycles toluene, and then cools it in a stainless steel tray while it is hot to obtain transparent glass-like solid sucrose benzoate.32L 0Kg, measured the average degree of substitution5 · 3, the utilization rate of sucrose96 · 1%, acyl chloride utilization96 · 4%.

[0027]Example 6

[0028]At500LAdd sucrose to the reactor.128Kg(373 · 9mol), water200Kg,48% sodium hydroxide aqueous solution

64Kg, stir and heat up to35,40 ℃, dissolve the light yellow transparent sucritol sodium aqueous solution and transferred to a high-level metering tank for later use. At1000LAdd benzoyl chloride to the reactor.305 · 9Kg(2177 · hoi), turn on the frozen salt water and control the material temperature.Ten,15 ℃Stir and add the above sucrose alcohol sodium aqueous solution to control the drop acceleration to keep the material temperature atTen,15 ℃As the reaction progresses, the system gradually thickens and slowly adds toluene.400Kg, to reduce viscosity; sucritol sodium aqueous solution about2One3The hourly drip is finished; then slowly drip through the high-level measuring tank.48% sodium hydroxide aqueous solution123 · 8Kg,Control the material temperature at the same time.Ten,15 ℃,PH8,1 1, add to slowly heat up to20,25 ℃, thermal insulation reaction2Hour. After the thermal insulation reaction, the heating rises to65,70 ℃, divide the water layer, and then add toluene.250KgUse water every time.50KgWash it with water and need to wash it.4Second (washing water can be used for the synthesis of the next batch of products) andPHIt is neutral. The

organic layer is transferred to500LThe reactor distillates and recycles toluene, and then cools it in a stainless steel tray while it is hot to obtain transparent glass-like solid sucrose benzoate.342 · 4Kg, measured the average degree of substitution5 · 8, the utilization rate of sucrose96 · 8%, acyl chloride utilization96 · 5%.

[0029]The raw materials used in the implementation of the production method of sucrose parabens are all industrial-grade and can be purchased in the domestic market.

[0030]The instruments and equipment used in the implementation of the production method of sucrose bens can be purchased from the domestic market according to the needs of chemical experiments, general reactors in production, liquid-liquid separation equipment, etc.

[0031]The invention is a small amount of wastewater produced in the production method of sucrose parabens, which has good biochemical properties and can be treated through the wastewater treatment system to meet the national discharge standard.

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Claims and claims1Page instructions4Page

(54) Name of invention

A method for preparing a sucrose parabenzate

(57) Summary

The invention discloses a method for preparing sucrose benzoate, which first reacts sucrose with sodium hydroxy oxide in water to obtain an aqueous solution of sodium sucralose. The resulting sodium aqueous

solution of sucral alcohol is added to the reaction, and a certain amount of toluene and sodium hydride aqueous solution continues to react completely. By adjusting the ratio of sucrose, benzoylchlor and sodium hydride, the average degree of substitution can be obtained in3,6Sucrose paramate with a specific degree of substitution between. The invention is stable due to the first production of a highly active sodium sucrose alcohol intermediate compared to the existing method.3,6Sucrose paraate with a specific degree of substitution between, and flatThe degree of substitution and the content of each component fluctuated less. Moreover, it is simple to operate, has high process stability, and the color of the product is low and stable, making it suitable for industrial production.

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Claims and claims 1 / 1

1.An average degree of substitution is3,6The preparation method of sucrose benzoate is characterized by the method described as: sucrose and sodium hydroxy oxideAReact in water to obtain an aqueous solution of sodium sucralose; sucrose and sodium hydroxygenAThe proportion of the amount of substances is1 : 1,3; Under continuous stirring, the aqueous solution of sodium sucralose is added to benzoylchlorine for reaction, and the temperature of the reaction solution is controlled as0,15℃During the reaction process, the reaction system becomes thick. When the reaction system is so thick that it is difficult to stir, toluene is added to the reaction system to reduce the viscosity of the reaction system. After the sodium sucral alcohol aqueous solution is dropped, sodium hydr oxide is added.BThe aqueous solution controls the temperature of the reaction solution0,15℃,pHValue8,1 1, after adding, it heats up to20,25 ℃, thermal

insulation reaction1,3hThe reaction is complete, and then warms up to65,70 ℃, separate the water layer, wash the organic layer with a small amount of water to neutral, and then distillate and recover toluene, and the rest is sucrose paraben; the proportion of the substance of sucrose, benzylchlor, and total sodium hydroxy oxide is1： 3,

6:3 ～ 6. 2。

2 ·According to the claim1The method described is characterized by the use of sucrose and sodium hydr oxideAWhen reacted in water, the amount of water is of sucrose quality0 · 5,3 倍。

3 ·According to the claim1The method is characterized by the fact that the total amount of toluene in the reaction system is sucrose quality3,8Double。

4·According to the claim1The method is characterized by washing the organic layer with water, and the mass dosage of the water is sucrose.1. 5,2Double.

5 ·According to the claim1The method is characterized by the ratio of the amount of the substance of sucrose and benzoylchlor to1： 3. 5,5 · 8.

6 ·According to the claim1The method is characterized by the ratio of the amount of the substance of sucrose and total sodium hydroxidation to1： 3 · 7,6. 1。

A method for preparing a sucrose parabenzate

(一) Technical field

[0001]The invention belongs to the chemical field, specifically a method for the preparation of sucrose parabens.

(二) Scenery technology

[0002]Sucrose parabenzate is used as a synthetic chemical raw material, coating and cosmetic raw material, and as a high-performance plastic modification additive for high-performance liquid crystal display film optical materials (which can give film-specific optical properties), holographic photosensitive materials, electronic image recording originals, as well as ribbons for thermal transfer printing, magnetic heat transfer printing, electro Different applications have corresponding requirements for the average replacement degree of sucrose parabens and the content of each component to achieve better use results.

[0003]U.S. patentUS3198784The ratio of substances reported to benzylchloride is1 : 6,Ten, the average degree of substitution of preparation is6,8Method of sucrose benzoate: benzoate reaction of sucrose in solvents such as benzene, toluene, metluene, ethylbenzene, chlorobenzene, chlorobenzene, monochloromethane, chloroform and carbon perchloride. After the reaction is completed, the average degree of substitution is6,8Of sucrose parabens. Japanese patent Zhao52One95625It is reported that the average degree of substitution in organic solvents such as ethylone, butyl ketone, dioxane, methyl aster asterate and tertyl alcohol with a certain solubility in water is1. 51,7 · 76The method of sucrose parabenzate.

[0004]First of all, the method described in the U.S. patent can only produce an average degree of substitution in6,8Of sucrose paraben, which cannot be prepared with an average degree of substitution3,6Of sucrose paraate, secondly, low utilization rate of sucrose and benzylchlor, low product yield, wastewaterCODHigher. Although the Japanese patent has synthesised the average degree of substitution1. 51,7 · 76Sucrose paraben, but this method has a large amount of water and organic solvent, and the solubility and low boiling point in the organic solvent water used, so there is a large amount of wastewater and wastewater per unit productChemical oxygen demandHigh, solvent consumption, high cost.

(三) Invention content

[0005]The technical problem to be solved by the invention is that the existence of existing technology cannot be replaced by3,6Sucrose parabens, low yield, high production costs, etc., or although an average degree of substitution can be produced3,6Of sucrose paraben, but there is a large amount of wastewater per unit product, wastewaterChemical

oxygen demandHigh, solvent consumption, high cost problems. The purpose of the invention is to provide a method that can not only produce a simple, effective and stable production of the average replacement degree in3,6Sucrose parabens with a specific degree of substitution, and high yield, low solvent consumption, low solvent residue in wastewater, low amount of wastewater per unit product and wastewaterChemical oxygen demandLow, low-impact, suitable for industrial production methods.

[0006]The technical scheme adopted by the invention is as follows:

[0007]A method for preparing sucrose benzoate. The method described is to combine sucrose with sodium hydroxy oxideAReact in water to obtain aqueous solution of sodium sucralol; under constant stirring, the aqueous solution of sodium sucralol is added to benzoylchlor for reaction, and the temperature of the reaction solution is controlled to0,15℃During the reaction process, the reaction system becomes thick. When the reaction system is so thick that it is difficult to stir, toluene is added to the reaction system to reduce the viscosity of the reaction system. After the sodium sucral alcohol aqueous solution is dropped, sodium hydr oxide is added.BThe aqueous solution controls the temperature of the reaction solution0,15℃,pHValue8,1 1, after adding, it heats up to20,25 ℃, thermal insulation reaction1,3hComplete reaction (optimal insulation reaction)2h), and then heat up to65,70 ℃, separate the aqueous layer, wash the organic layer with a small amount of water to neutral, and then distillate and recover toluene, and the residue is sucrose paraben; the substances described in sucrose, benzylchlor, and total sodium hydr oxide

The quantity ratio is1： 3,6： 3,6 · 2. The average replacement degree of the sucrose parabens is3,6.

[0008]In the method of the invention, sodium hydr oxideAWith sodium hydrideBSodium hydride is added in different steps, using sodium hydrideAWith sodium hydrideBDistinguish, sodium hydrideAWith sodium hydrideBThe total amount is the total amount of sodium hydride. Preferable, the sucrose and sodium hydr oxideAThe proportion of the amount of substances is1： 1,3, which is preferred as1： 2,2 · 6.

[0009]The ratio of the amount of the substance of sucrose and benzoylchlor is preferred as1： 3 · 5,5 · 8.

[0010]The ratio of the amount of the substance of sucrose and total sodium hydr oxide is preferred as1： 3 · 7,6 · 1.

[0011]The invention describes the use of sucrose and sodium hydroxyAReacting in water, the amount of water is usually the quality of sucrose0 · 5,3, times

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[0012]The organic layer is washed neutral with a small amount of water, and the amount of water usually is sucrose-quality1 . 5,2Double,可将Water is added many times for washing, making it easy to wash effectively. And the washed water can also be used for the next batch of synthetic water or for washing.

[0013]In the reaction system, as the reaction continues to proceed, the viscosity of the reaction system increases, which increases the difficulty of stirring the mixed reaction raw materials. Therefore, toluene is needed to reduce the viscosity of the reaction system, facilitate full mixing of the mixing reaction raw materials, and prevent excessive substitution caused by uneven dispersion of raw materials. Monitor the reaction system during the reaction process, according to the needs of the reaction system,

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hen the reaction system is thick enough to stir, toluene is added. Usually, the amount of toluene added to the reaction system is sucrose quality.38Double.

[0014]The reaction principle of the invention is that sucrose is first reacted with a certain amount of sodium hydroxy oxide in water to obtain aqueous solution of sodium sucralose, which improves the reaction activity of sucrose. When sodium sucralate aqueous alcohol is added to the benzoylchlor reaction, benzoylchlor plays the1Someone2Replaced sucrose parabens, as the reaction progresses, add an appropriate amount of toluene to dissolve the reaction2One3Replace the sucrose paraben and reduce the viscosity of the system to facilitate2One3The replaced sucrose parabens further react with benzylchloride to produce the target product. Therefore, the average substitution degree of preparation can be achieved by adjusting the ratio of sucrose and benzoylchlor.3,6The purpose of the specific degree of substitution between sucrose parabens.

[0015]The invention has the following significant advantages compared to existing methods:1, because a highly active sodium sucralol intermediate was first made, it can be stable by adjusting the ratio of sucrose and benzoylchlorine.3,6Sucrose parabens with a specific degree of substitution between them, and the average degree of substitution and the content of each component fluctuate little.2, simple operation, high process stability, low color and stable product, suitable for industrial production.3, due to improved reaction activity and homogeneity, the utilization rate of sucrose and benzylchlor has been improved.4, low solvent consumption and less wastewater, reducing production costs while reducing the impact on the impact on the company.

(4) Specific implementation methods

[0016]The invention is further described in combination with the embodiment below, but the scope of protection of the invention is not limited to this. Any simple modification, equivalent change or modification of the embodiment based on the technical substance of the invention shall fall within the scope of protection of the invention.

[001 7]Example 1 of implementation

[0018]At250m1Add sucrose to the conical bottle40 · Og 0 16 · 8mmo l), water60m1,48% sodium hydride aqueous solution20g, in35,40 ℃Mix and dissolve the light yellow transparent sucrose sodium aqueous solution and transfer it into a constant pressure drop water funnel for use. In an ice bath, a stirrer, a thermometer and the aforementioned constant pressure drip funnel500m1Four-neck bottle, add benzyl chlorine57 · 9g(412 · 1" (1), control the temperature of the material5,10℃Add the above sucralate sodium aqueous solution. As the reaction progresses, the material gradually thickens and slowly adds toluene180m1To reduce viscosity; sucral alcohol sodium aqueous solution about30After adding the drops for minutes, then add them to the constant pressure droplet funnel48% sodium hydride aqueous solution16g, add it to the reaction system48% sodium hydr oxide aqueous solution while controlling the temperature of the material5,10℃,PI-18,1 1, Gabbi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time20m1, wash4(Washing water is used for the next batch of synthesis or water washing). After distillation and recovery of toluene, pour it into a stainless steel tray while it is hot, and cool it to obtain a transparent glass-like solid sucrose paraben79 · l g, measured average degree of substitution3 · 5, sugar utilization95 · 8%, acylchloride utilization rate95 · 1%.

[0019]Example 2 of implementation

[0020]At250m1Add sucrose to the conical bottle40 · Og(116 · 8" (1), water50ml,48% sodium hydride aqueous solution25g, in35,40 ℃Mix and dissolve the light yellow transparent sucrose sodium aqueous solution and transfer it into a constant pressure drop water funnel for use. In an ice bath, a stirrer, a thermometer and the aforementioned constant pressure drip funnel500m1Four-neck bottle, add benzyl chlorine74 · 4g(529 · 5"(1), control the temperature of the materialTen,15℃Add the above sucralate sodium aqueous solution. As the reaction progresses, the material gradually thickens and slowly adds toluene200mLTo reduce viscosity; sucrose sodium aqueous solution about40After adding the drops for minutes, then add them to the constant pressure droplet funnel48% sodium hydride aqueous solution21. l g,Add to the reaction system48% sodium hydr oxide aqueous solution while controlling the temperature of the materialTen,15℃,PH8,1 1, Gabbi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time20m1, wash3(Washing water is used for the next batch of

synthesis or water washing). After distillation and recovery of toluene, pour it into a stainless steel tray while it is hot, and cool it to obtain a transparent glass-like solid sucrose paraben91. l g, measured average degree of substitution4 · 5, sugar utilization96 · 2%, acylchloride utilization rate95 · 5%.

[0021]Example 3 of implementation

[0022]At250m1Add sucrose to the conical bottle32 · Og(93 · 5mmol), water50m1,48% sodium hydride aqueous solution22 · 5g, in35,40 ℃Mix and dissolve the light yellow transparent sucrose sodium aqueous solution and transfer it into a constant pressure drop water funnel for use. In an ice bath, a stirrer, a thermometer and the aforementioned constant pressure drip funnel500m1Four-neck bottle, add benzyl chlorine76 · l g(542 · Immol), control the temperature of the material0,5℃Add the above sucralate sodium aqueous solution. As the reaction progresses, the material gradually thickens and slowly adds toluene200m1To reduce viscosity; sucral alcohol sodium aqueous solution about30After adding the drops for minutes, then add them to the constant pressure droplet funnel48% sodium hydride aqueous solution25g, add it to the reaction system48% sodium hydr oxide aqueous solution while controlling the temperature of the material0,5℃,PH8,1 1, Gabbi, slowly heat up the material to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises to65,70 ℃, separate the water layer, wash the organic layer to neutral, and use water every time20ml,Rinsing3(Washing water is used for the next batch of synthesis or water washing). After distillation and recovery of toluene, pour it into a stainless steel tray while it is hot, and cool it to obtain a transparent glass-like solid sucrose paraben85 · Og,Measured average degree of substitution5 · 7, sugar utilization97 · 2%, benzyl chlorine utilization rate95 · 6%.

[0023]Example 4 of implementation

[0024]At500LAdd sucrose to the reactor128Kg(373 · 9mol), water200Kg,48% sodium hydride aqueous solution

64Kg, stir to heat up to35,40 ℃, dissolved into a light yellow transparent sucal alcohol sodium aqueous solution and transferred to a high-level metering tank for use. At1000LAdd benzylchloride to the reactor278 · 0Kg 0978 · 9mol), freeze salt water to control the temperature of the material5,10℃, stir the drops and add the above-mentioned sucralol sodium aqueous solution to control the drop acceleration to keep the material temperature5,10℃As the reaction progresses, the system gradually thickens and slowly adds toluene400KgTo reduce viscosity; sucral alcohol sodium aqueous solution about2One3The hour drops are finished; then slowly add them through high-level metering tanks48% sodium hydride aqueous solution107 · 2Kg,Simultaneously control the material temperature5,10℃,PH8,1 1, Gabbi slowly warmed up to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises

to65,70 ℃, separate the water layer and add toluene200Kg, and use water every time50KgWash with water, need to wash4(Washing water is used for the synthesis of the next batch of products) and toPHNeutral. It is neutral. The organic layer is transferred to500LThe reactor distils and recovers toluene, and then puts it in a stainless steel tray while it is hot to cool and mold to obtain transparent glass-shaped solid sucrose parabens32L 2Kg, measured average degree of substitution5 · 3, sugar utilization96 · 1%, acylchloride utilization rate96 · 3%.

[0025]Example 5 of implementation

[0026]At500LAdd sucrose to the reactor128Kg(373 · 9mol), Implementation Example 4 Recovered Washing Water200Kg,48% sodium hydride aqueous solution64Kg, stir to heat up to35,40 ℃, dissolved into a light yellow transparent sucal alcohol sodium aqueous solution and transferred to a high-level metering tank for use. At1000LAdd benzylchloride to the reactor277 · 5Kg0975 · Omi),Open frozen salt water to control the temperature of the material5,10℃, stir the drops and add the above-mentioned sucralol sodium aqueous solution to control the drop acceleration to keep the material temperature5,10℃As the reaction progresses, the system gradually thickens and slowly adds toluene400KgTo reduce viscosity; sucral alcohol sodium aqueous solution about2One3The hour drops are finished; then slowly add them through high-level metering tanks48% sodium hydride aqueous solution106 · 5Kg,Simultaneously control the material temperature5,10℃,PH8,1 1, Gabbi slowly warmed up to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises to65,70 ℃, separate the water layer and add toluene200Kg, and use water every time50KgWash with water, need to wash4(Washing water is used for the synthesis of the next batch of products) and toPHNeutral. It is neutral. The organic layer is transferred to500LThe reactor distils and recovers toluene, and then puts it in a stainless steel tray while it is hot to cool and mold to obtain transparent glass-shaped solid sucrose parabens32L 0Kg, measured average degree of substitution5 · 3, sugar utilization96 · 1%, acylchloride utilization rate96 · 4%.

[0027]Example 6 of the implementation

[0028]At500LAdd sucrose to the reactor128Kg(373 · 9mol), water200Kg,48% sodium hydride aqueous solution

64Kg, stir to heat up to35,40 ℃, dissolved into a light yellow transparent sucal alcohol sodium aqueous solution and transferred to a high-level metering tank for use. At1000LAdd benzylchloride to the reactor305 · 9Kg(2177 · hoi), freeze salt water to control the temperature of the materialTen,15℃, stir the drops and add the above-mentioned sucralol sodium aqueous solution to control the drop acceleration to keep the material temperatureTen,15℃As the reaction progresses, the system gradually thickens and slowly adds toluene400KgTo reduce viscosity; sucral alcohol sodium aqueous solution about2One3The hour drops are finished; then slowly add them through high-level metering tanks48% sodium hydride aqueous solution123 · 8Kg,Simultaneously control the material temperatureTen,15℃,PH8,1 1, Gabbi slowly warmed up to20,25 ℃, thermal insulation reaction2Hours. After the thermal insulation reaction, the heat rises to65,70 ℃, separate the water layer and add toluene250Kg, and use water every time50KgWash with water, need to wash4(Washing water can be used to 合成 the

next batch of products) andPHNeutral. It is neutral. The organic layer is transferred to500LThe reactor distils and recovers toluene, and then puts it in a stainless steel tray while it is hot to cool and mold to obtain transparent glass-shaped solid sucrose parabens342 · 4Kg, measured average degree of substitution5 · 8, sugar utilization96 · 8%, acylchloride utilization rate96 · 5%.

[0029]The raw materials used in the implementation of a production method of sucrose parabens are of industrial grade and can be procured in the domestic market.

[0030]The instruments and equipment used in the implementation of a production method of sucrose parabens are chemical experiments, general reactors in production, liquid-liquid separation equipment, etc. can be purchased from the domestic market as needed.

[0031]A small amount of wastewater produced in the production method of sucrose parabenzate has good biochemical properties and can be treated through the wastewater treatment system to meet the national discharge standard.