Research Report

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In the past two days, I have looked up the patents of Adanas and reviewed them. In the next two days, I will consult and study the equipment-patents of Adanas.

A preparation method of sucrose fatty acid ester with low ash content

(CN 106243166 B)

Sucrose ester is a kind of green Nonionic surfactant with sucrose as hydrophilic group and fatty acid chain as hydrophobic group with good biodegradability, so it can be widely used in food additives, detergents, detergents and cosmetics and has potential applications in pharmaceutical, biochemical and biomedical fields. The raw materials of sucrose fatty acid esters are cheap and easily available renewable resources with widely controllable hydrophilic and lipophilic balance. In addition to good emulsifying properties, sucrose esters have complete biodegradability and are environmentally friendly, non-toxic, skin compatible, odorless and odorless. However, in the process of application of production technology, sucrose ester is generally synthesized by transesterification of fatty acid methyl ester with sucrose in organic solvent under the catalysis of alkaline catalyst and heating. This synthesis method has high solvent toxicity and high cost, which will inevitably bring some restrictions to the application of sucrose ester synthesized by solvent method. The current production process uses solvent-free method to produce sucrose fatty acid esters, which can solve the adverse effects of solvent toxicity and has been widely used as a food additive. In the solvent-free method, the mixed catalytic reaction of alkaline catalyst and reactant results in a high ash content of the product, and the ash composition of sucrose fatty acid ester product is mainly composed of alkaline catalyst and metal ions. This is because the esterification products of the existing technology contain sucrose esters and fatty acids, as well as ash impurities alkaline catalysts and metal ions. However, there is no good method to remove these ash impurities, which results in high ash content and poor quality of the final sucrose fatty acid ester. The existence of these alkaline substances is not conducive to the application of products and physical health. The application range of sucrose fatty acid ester with high ash content is getting narrower and narrower, and it is about to face the fate of elimination. For example, the foreign Nestle company clearly proposed that the ash content of sucrose fatty acid esters purchased must be less than or equal to 1.5%. The existing methods for the preparation of sucrose fatty acid esters have the following defects: 1. The ash content of sucrose fatty acid ester is high, which needs to be improved. 2. The esterification degree of the product is low, the purity of sucrose fatty acid ester is low, and the production process needs to be improved. It is urgent to produce low ash and high quality sucrose fatty acid esters to meet the market demand. The invention discloses a preparation method of low ash sucrose fatty acid ester, which comprises the following steps: a, fatty acid methyl ester and part of sucrose powder are esterified under the action of alkaline catalyst, and after the reaction is complete, water equivalent to 0.4 times the volume of the product after the reaction is diluted, and then the diluent is passed through an ion exchange column to remove metal ions. The alkaline catalyst is alkaline metal salt or metal hydroxide; B, after the ion exchange is finished, the water of the material which has completed the ion exchange is removed, and after the water is removed, the sucrose powder is added to continue the reaction. The invention has the advantages of low cost and convenient operation, and can solve the problems of low esterification degree of products, low purity of sucrose fatty acid esters and high ash content of products in the prior art.

5L fatty acid methyl ester.

Reaction kettle.

1.1L potassium hydroxide aqueous solution with 5mol/L concentration

Vacuum to-0.1Mpa

The steam was heated to 127 ℃

3.2kg sucrose powder.

127 ℃. 1.5h

Condensation. Cool

(the exchange column temperature is kept at 70 ℃ and the flow rate is controlled at 150mL/min)

Methanol receiver

6L water .Heat to 70 ℃

Strong acid ion exchange column

(removal of potassium ion)

The water was removed by vacuum heating at 110 ℃.

Add 300g sucrose powder.

Add 300g sucrose powder.

The low ash sucrose fatty acid ester

Synthesis of sucrose ester by fixed-bed reactor with alkali catalyst

(CN 104804050 B)

Sucrose ester is a kind of green Nonionic surfactant with sucrose as hydrophilic group and fatty acid chain as hydrophobic group with good biodegradability, so it can be widely used in detergents, detergents and cosmetics and has potential applications in pharmaceutical, biochemical and biomedical fields. The raw materials of sugar fatty acid esters are cheap and easily available renewable resources with widely controllable hydrophilic and lipophilic balance. In addition to good emulsifying properties, sucrose esters have complete biodegradability and are environmentally friendly, non-toxic, skin compatible, odorless and odorless. The earliest synthesis of sucrose esters dates back to the mid-19th century, from then until 1956, sucrose esters were either directly esterified with sucrose and fatty acids or anhydride, or chloroacetate with fatty acyl chloride in pyridine. These synthesis methods are either low yield, high solvent toxicity and high cost, so it is difficult to realize industrialization. The specific synthesis methods of these early sucrose esters mostly choose organic solvents which can dissolve sugar and oil at the same time, such as dimethyl alum, dimethylacetamide, dimethylformamide and so on. Sugar and fatty acids are transesterification in the presence of alkaline catalysts. the usual alkaline catalysts are sodium hydroxide and potassium hydroxide. However, in the process of application, sucrose ester is generally synthesized by transesterification of methyl stearate with sucrose in organic solvent under the catalysis of alkaline catalyst and heating. However, organic solvents such as dimethylacetamide, dimethylformamide and so on have certain toxicity, which will inevitably limit the application of sucrose ester synthesized by solvent method. At the same time, the mixed catalysis of alkaline catalyst and reactants will bring further difficulties to the subsequent purification of the product, resulting in higher ash content of the product and poor recycling ability of the catalyst. The existing process for the synthesis of sucrose ester is by mixing reactants and catalysts and steaming the products from the reaction system in the reaction process. This method belongs to intermittent production, with long production cycle, high production cost and low product conversion rate, so it is not suitable for large-scale industrial production.

The process is a process for the synthesis of sucrose ester using alkali catalyst fixed bed reactor, which includes the following steps: meta titanic acid (also known as hydrated titanium dioxide) produced by sulfuric acid method is used as the starting material, the raw material is purified by adjusting pH value, washing and filtration, dehydrated and dried, crushed, screened and packaged to become the final product. The preparation method and material are simple, and the prepared nano-titanium dioxide has large specific surface area and high TiO2 content.

600g methyl stearate.

↓ 100 ℃. Melt.

Alkali catalyst fixed bed reactor

↓ The flow rate of gear pump is 18.3L/min. 140 ℃.

↓ Sucrose powder 225.9g. (the average amount of sucrose added per 10min is 12.6 g, for a total of 5 hours)

Sucrose ester

↓ Wash it many times. Drying under reduced pressure.

Pure sucrose ester.
The yield was 73.2%.

the reactor is connected to the vacuum distillation unit during the reaction to keep the reaction system at negative pressure, and the methanol produced by the reaction is collected at the end of the distillation unit

Preparation method of sucrose ester

(CN 103396460 B)

There are four main methods for the synthesis of sucrose esters: solvent method, micro emulsification method, solvent-free method and enzyme catalytic method. DMF or DMSO are used as solvents in solvent method, but these two solvents are toxic, which limits the application of sucrose esters in food and other industries. In the micro emulsification method, propylene glycol or water is used instead of the toxic solvent used in the solvent method, and the emulsifier is added to make the reaction system approximate to a homogeneous system. The solvent-free rule is to make the molten phase homogeneous and the reaction stable by adding emulsifier or surfactant to the reaction system. However, in general, the reaction temperature of the solvent-free method is high, the reaction is not easy to carry out, the yield is low, and the product quality can not be guaranteed. At present, the solvent-free method usually uses alkali metal hydroxide such as potassium hydroxide or sodium hydroxide as the catalyst, and the residual phenomenon of the catalyst in the product is serious, which strictly limits its application in the food industry. the subsequent separation and purification steps are more tedious and easy to produce a lot of wastewater. The invention adopts heterogeneous catalyst calcium oxide, which is used as a catalyst for heterogeneous catalytic transesterification reaction, and has been widely used in the field of biodiesel. After pretreatment, the calcium oxide has a strong specific surface area and has good stability. The catalytic life is long and can be used continuously for several times, so it is valued by more and more chemists and chemical experts.

The invention relates to a method for preparing sucrose ester, belonging to the technical field of Nonionic surfactant. In this method, a small amount of water and free fatty acids in fatty acid methyl ester are removed, and then the solid catalyst is added to the fatty acid methyl ester after water removal, and sucrose is added in a certain proportion under the condition of uniform stirring. In the prior art, alkali metal hydroxides are often used as catalysts in the preparation of sucrose esters in industry, which is easy to cause tedious purification steps of subsequent products, resulting in a large amount of waste water. The invention adopts alkaline earth metal oxides commonly used in industry as solid catalysts, the reaction conditions are mild, the solid catalysts will not remain in the product and can be reused, and are environmentally friendly and reduce costs.

4g calcium oxide (water remover).
30g methyl stearate.

↓ Stir

↓ A three-neck bottle
51g sucrose

↓ 3.2 g calcium oxide (catalyst).
Stir. 30min .

↓ 130 ℃.4 h
Atmospheric pressure or decompression

↓ Liquid separation funnel

Sucrose ester.

↓ 80 ℃. Vacuum distillation

Upper oil phase