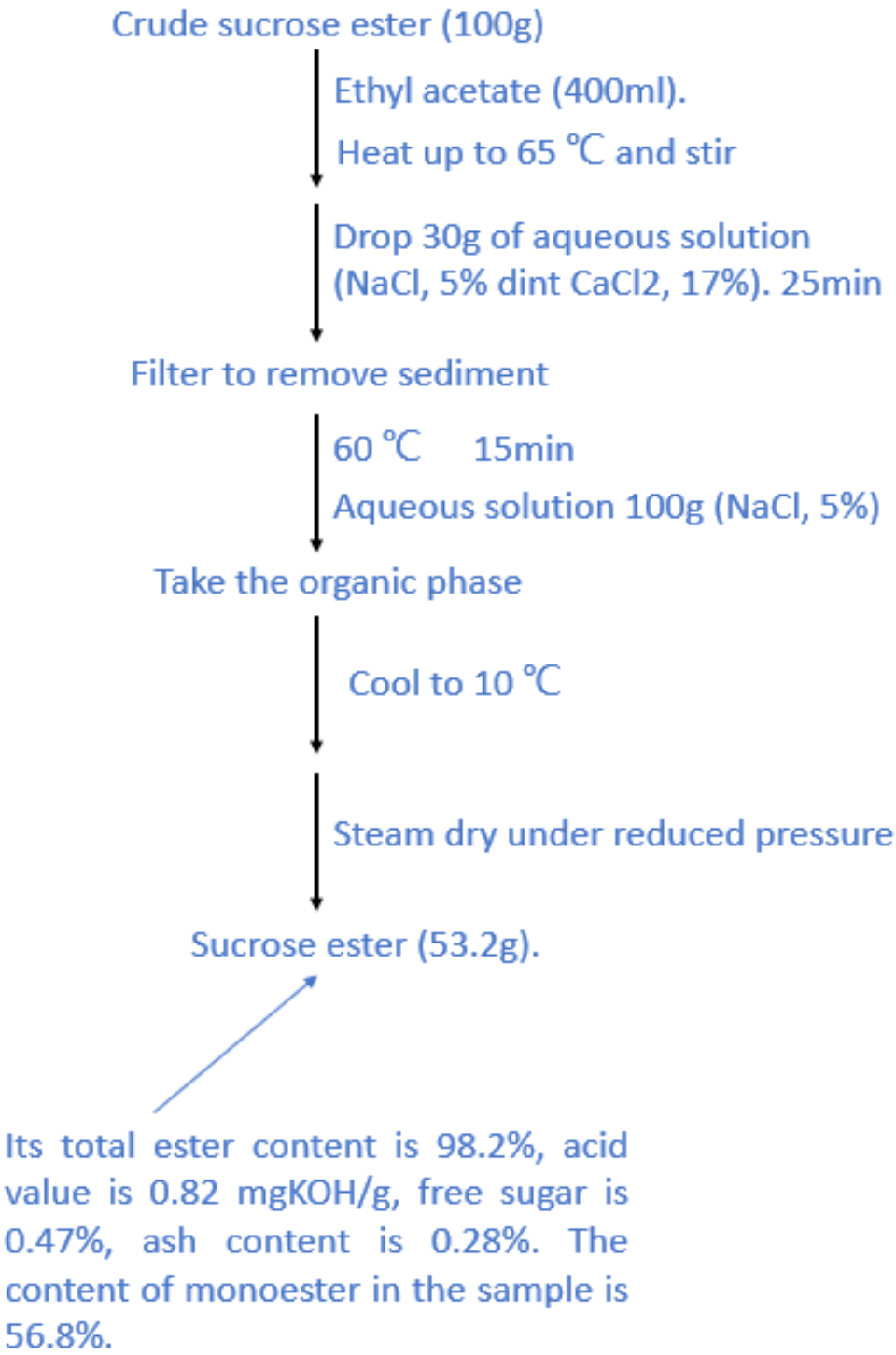
4-share A method for purification of sucrose ester (CN 103087118 B)

At present, most of the industrial methods for the synthesis of sucrose esters are transesterification, which can be divided into solvent method and solvent-free method. Sucrose esters 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 the purity of the product is not high. The invention patent no. CN 200510006011.l discloses a method for purifying sucrose fatty acid esters, which is suitable for the purification of crude sucrose esters produced by transesterification, but the sucrose ester products purified by this method have a maximum total ester content of only 94%, and the total ester content is still low, which can not meet the higher requirements of users for product purity.

This process is a purification method of sucrose fatty acid ester. The method is that the crude sucrose ester is dissolved in an organic solvent that can be separated from water, and the crude sucrose ester solution is obtained, and the salt aqueous solution dissolved with alkaline earth metal salt and / or alkaline earth metal oxide is added under stirring. the fatty acid soaps in the crude sucrose ester solution produce fatty acid alkaline earth metal salts insoluble in organic solvents, then remove the resulting solid fatty acid alkaline earth metal salts, and then add salt water for stirring extraction. The lower water layer is separated statically, the supernatant is cooled to 0 ℃-40 ℃ under stirring, the sucrose ester crystal is precipitated in the form of precipitation, the liquid is removed and the solid material is removed, and the sucrose ester product is obtained after drying. The process introduces the process of cooling crystallization, removes the residue of unreacted raw materials such as fatty acid methyl ester in the product, and further improves the content grade of the product, and the total ester content in the product is more than 98%.


Crude sucrose ester (100g)
Ethyl acetate (400ml).
Heat up to 65 ℃ and stir
Drop 30g of aqueous solution
(NaCl, 5% dint CaCl2, 17%). 25min
Filter to remove sediment
60 ℃ 15min
Aqueous solution 100g (NaCl, 5%)
Take the organic phase
Cool to 10 ℃
Steam dry under reduced pressure
Sucrose ester (53.2g).
Its total ester content is 98.2%, acid value is 0.82 mgKOH/g, free sugar is 0.47%, ash content is 0.28%. The content of monoester in the sample is 56.8%.

5-share A method for purification of sucrose ester (CN 103087118 B)

This process is a production method of sucrose ester with low HLB value and its application in the preparation of special antioxidants for oil and fat. The production method of this process includes the following steps: (1) adding tea seed oil, methanol and concentrated sulfuric acid to synthesize camellia seed oil fatty acid methyl ester, (2) adding KOH solution, stearic acid, sucrose, catalyst, palmitic acid methyl ester and camellia seed oil fatty acid methyl ester to prepare sucrose fatty acid ester with high degree of substitution by transesterification. Then the sucrose fatty acid ester was separated and purified by adding anhydrous ethanol reflux, and then the catalyst and unreacted raw materials were removed with petroleum ether to get the available sucrose ester. The sucrose ester with low HLB value prepared by this process can be mixed with vitamin E, sage acid or tea polyphenol to produce natural oil antioxidants with excellent performance, which can replace the chemical synthetic oil antioxidants that will be banned soon.

10 grams of KOH. 200ml of water. 50 grams of stearic acid.

Stir. 70 ℃.

350 grams of sugar. 35 grams of potassium carbonate. 150g methyl palmitate. 600 grams of fatty acid methyl ester of camellia seed oil

Stir at 80 ℃ for 30 minutes

Stir at 80 ℃ for 30 minutes
rose to 125 ℃. Pressure-0.098MPa.
The reaction time is 6 hours

cooling to 90-95 ℃
add 1500 ml of anhydrous ethanol
Reflux for 20 minutes

Add glacial acetic acid to adjust the PH value about 7.0
Filter while it's hot

Filter cake

1500 ml anhydrous ethanol.
78.5 ℃ and 90 ℃ . Extraction.
Filter.

Extractive liquid

Ethanol was evaporated at 78.5-90 ℃
Petroleum ether was washed twice
Remove petroleum ether

Remove petroleum ether

The crude yield is about 76%. GPC analysis results: monoester 18.7%, diester 28.3%, triester 25.6%, tetraester and above 19.4%, HLB value = 3, softening point 58 ℃.

6-share A method for analyzing the Distribution of various Esters in Sucrose Fatty Acid Esters by Gel Permeation Chromatography (CN 1030134872 B)

China promulgated the standard of sucrose ester (GB8272-1987) as early as 1987, stipulated the standard method for the quality analysis of sucrose ester, and replaced the old standard with the new standard (GB8272-2009) in 2009 to further improve the quality control system of sucrose ester. The standard stipulates the determination methods for the appearance, composition, acid value and free sugar of sucrose ester, but it is mainly the traditional volumetric analysis method, and the analysis of the composition is limited to qualitative analysis. The application of modern analytical instruments and the newly developed analytical methods in recent years are not reflected in the standard. Thin layer chromatography (TLC) is widely used in the qualitative and quantitative analysis of sucrose esters, but because sucrose esters themselves do not show color, it is necessary to use chromogenic agents. Gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) are also used to quantitatively and qualitatively analyze sucrose esters, but the derivation of sucrose esters is tedious. As a rapid quantitative method, high performance liquid chromatography (HPLC) ultraviolet detection and high performance liquid chromatography differential detection have been used for the analysis of sucrose esters. Because the ultraviolet absorption of sucrose esters is weak, the differential detector can not be gradient eluted. Therefore, it is of great significance to establish a general and rapid method for the analysis of sucrose esters in order to control the reaction end point, optimize the reaction process and monitor the quality of the products.

This process is a method to analyze the distribution of various esters in sucrose fatty acid esters by gel permeation chromatography. In this method, the reference substance of sucrose fatty acid ester was dissolved with tetrahydrofuran to prepare the reference solution, and the reference solution was analyzed by gel permeation chromatography. According to the GPC spectrum, the chromatographic peak area values of various esters in the control substance were obtained, the composition of various esters was calculated by area normalization method, and the retention time of various esters was obtained. Then, the sample of sucrose fatty acid ester was dissolved in tetrahydrobaran to prepare the sample solution, and the GPC spectrum of the sample was obtained under the same chromatographic conditions. According to the retention time of various esters in the reference substance, the peak area value of various esters in the sample was obtained, and the distribution of various esters in sucrose fatty acid esters was calculated by area normalization method. This process has the advantages of simple sample treatment method, simple operation, short detection time, good reproducibility, high precision and simultaneous determination of various esters in sucrose fatty acid esters.

1.Gel permeation chromatography and differential refractive detector were used to analyze the sucrose fatty acid ester reference solution one by one, and the GPC spectra were obtained.

2.The specific analysis process of the retention time of various esters in the reference substance of sucrose fatty acid esters is as follows.

Taking the reference substance of sucrose fatty acid ester with HLB value of 11 as an example, the chromatographic peak areas of the three esters of the reference substance were calculated according to the GPC spectrum 。

The composition of the three esters (i.e. the relative content of the three esters) was calculated by the area normalization method.

Refer to the composition of three esters in the reference substance of sucrose fatty acid ester with HLB value = 11 (triester 10%, diester 31%, monoester 59%)

correspond to the peak height and retention time of triester, diester and monoester in the test sample.