

Research Report

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In the past two days, I have completed the study of three patents of Gaotong on the synthetic process of sucrose esters, and summarized these three patents. In the next two days (before Thursday), I will look for patents related to the anhydrous synthesis of sucrose esters and review them.

Gaotong01 A method of preparing Sucrose Fatty Acid Ester particles by spray cooling

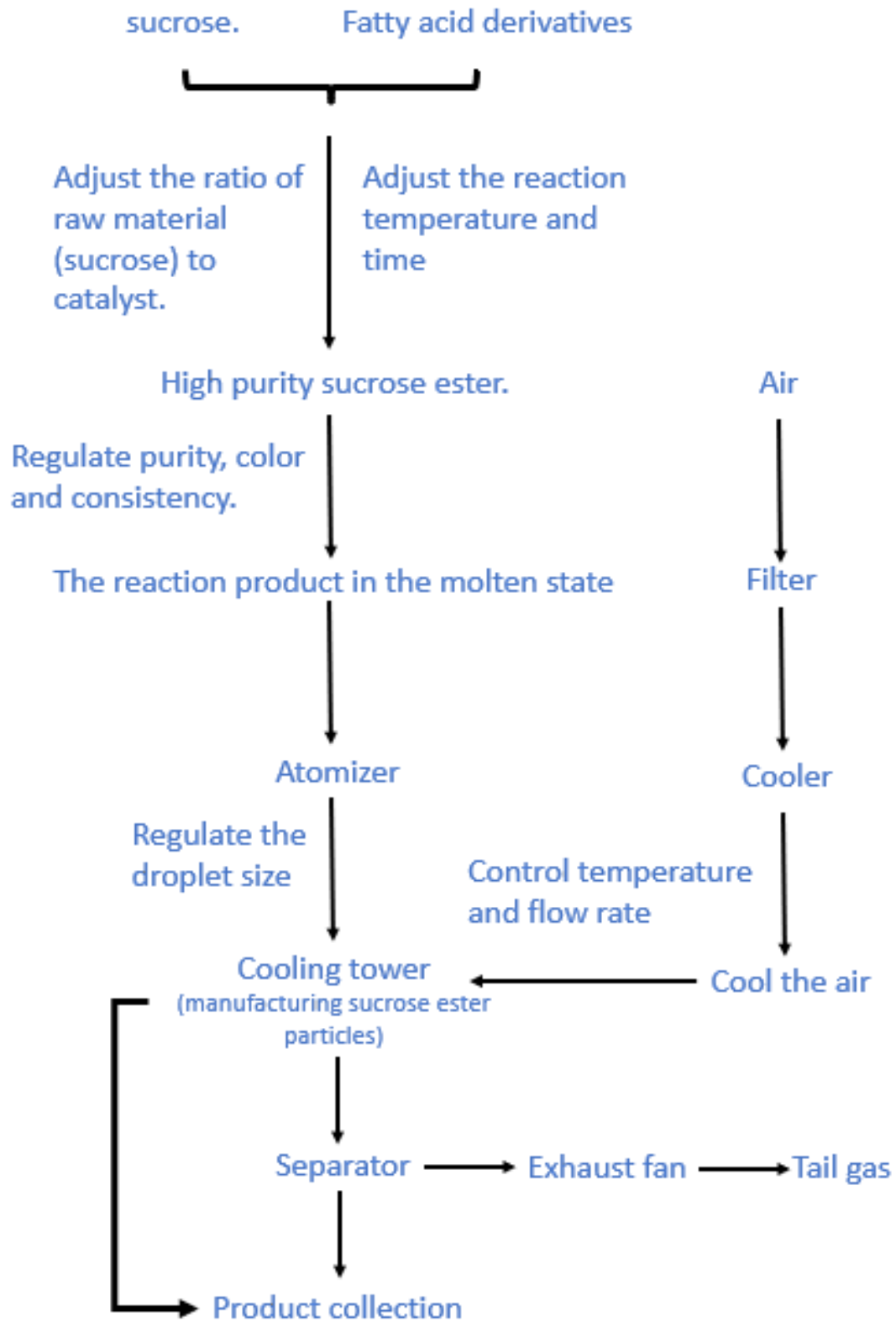
(CN 108067160 A)

At present, the main production method of sucrose fatty acid esters is solvent-free method, and the production law of solvent-free sucrose fatty acid esters is to form sucrose fatty acid esters by esterification of sucrose and fatty acid esters in the melting phase at high temperature. the reaction products of traditional sucrose fatty acid esters need to be naturally cooled into bulk or waxy solids, and then mechanically crushed into powdered products. In addition, due to the moisture absorption of sucrose, especially the excessive content of reducing sugar, sucrose fatty acid esters are easy to absorb moisture and agglomerate in the process of natural cooling and mechanical crushing of sucrose fatty acid esters, as well as during the storage of finished products; therefore, the traditional solventless sucrose fatty acid ester production process, especially the post-processing process, seriously affected the appearance quality and performance of sucrose fatty acid ester products. In addition, the traditional mechanical grinding process also produces a large amount of dust, which may pollute the environment and products, and may also have an impact on the health of direct production personnel.

The synthesis process is a method for preparing sucrose fatty acid ester particles by spray cooling. In this method, the molten sucrose fatty acid ester produced by the solvent-free method is sprayed directly in the cooling tower through the atomizer, and the finished sucrose fatty acid ester particles are solidified after heat exchange with the air in the cooling tower. This process is completely different from the traditional production process of sucrose fatty acid esters, and the reaction products do not need to be naturally cooled or mechanically crushed, which makes the production of sucrose fatty acid esters from feeding, reaction to the final product. the whole production process is completed at one time in a fully closed environment to avoid environmental pollution, product pollution and human harm caused by dust, and greatly improve the quality of sucrose fatty acid esters. Compared with the traditional powdered sucrose fatty acid ester processed by traditional mechanical pulverization, the product produced by this process has the advantages of uniform particle size distribution, high sphericity, good fluidity, not easy to moisture, high wettability and solubility.

In my opinion, the two key points of this process are **the reaction conditions and cooling conditions** in the molten state. The specific conditions for the production of melted sucrose fatty acid esters by solvent-free method are as follows: the mass ratio of raw material sucrose to fatty acid esters is 1. 4% 6, the amount of catalyst is 1% 5% of the total mass of raw materials, the whole reaction system should be controlled in a vacuum state where the vacuum degree is greater than 0.094MPa, the reaction temperature should be controlled at 120 °C ~ 140 °C, and the reaction time should be controlled at 3 °C for 6 hours. The spray cooling conditions of the atomizer are as follows: the speed of the atomizer is 2000~12000r/min, the inlet air temperature is 5 °C

20 °C, the outlet air temperature is 30 °C 45 °C, the feed temperature is 100 °C 130 °C, the feed speed is 30~60OL/h, and the cold air flow rate is 100 cubic meters per hour.



Gaotong02 A method for preparing Sucrose Ester particles with drying and forming

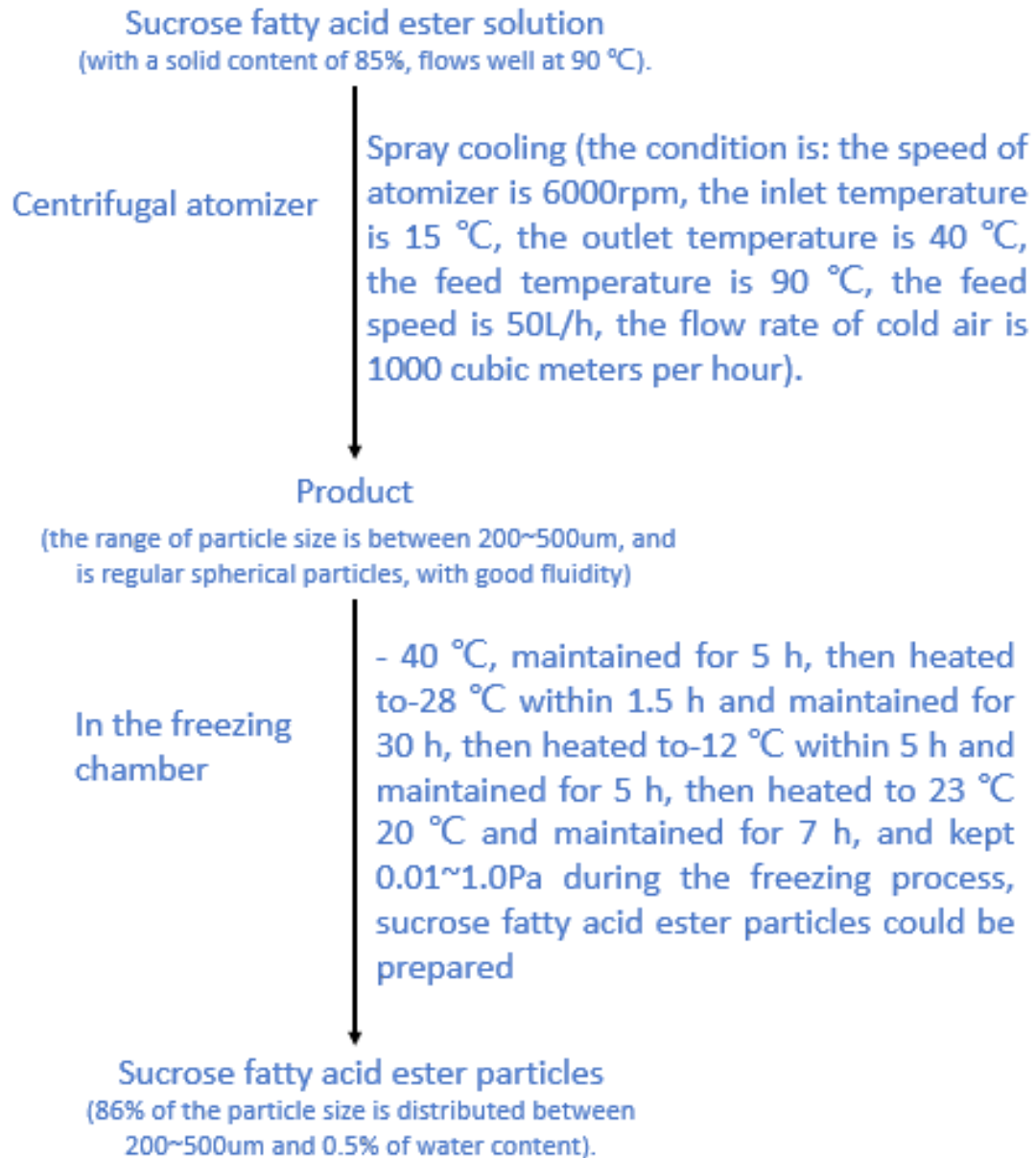
(CN 108067159 A)

At present, there are four main methods for the synthesis of sucrose fatty acid esters, namely, solvent method, micro-emulsification method, solvent-free method and enzyme catalytic method. No matter what method is used to produce sucrose fatty acid esters, the reaction products of sucrose fatty acid esters are either low in the target product, or leave a large number of impurities, or even some potentially toxic impurities. These impurities make the reaction products of sucrose fatty acid esters often can not meet the stringent requirements of food, medicine, cosmetics and other fields, can not be used directly, and need further separation and purification in order to expand the application field and scope of sucrose fatty acid esters. In addition, due to the moisture absorption of sucrose, sucrose fatty acid esters usually need to be dried after separation and purification, and then crushed to become the traditional powdered sucrose fatty acid esters. Therefore, the traditional production process of solvent-free sucrose fatty acid esters, especially the post-processing process, has seriously affected the appearance quality and performance of sucrose fatty acid esters. In addition, the traditional mechanical grinding process also produces a large amount of dust, which may pollute the environment and products, and may also have an impact on the health of direct production personnel.

This process is a preparation method of sucrose fatty acid ester particles which integrates drying molding. In this method, the reaction product of sucrose fatty acid ester was separated and purified, and then it was sprayed in the cooling tower by atomizer, and then the sucrose fatty acid ester particles were obtained by heat exchange with the air in the cooling tower. finally, the volatile components were removed by freeze-drying to obtain sucrose ester particles. It is completely different from the traditional production process of sucrose fatty acid ester. The production of sucrose fatty acid esters involved in this process does not need to be dried and then cooled, and finally crushed, but after separation and purification, drying is integrated and completed in a fully enclosed environment through spray freeze-drying process. avoid environmental pollution, product pollution and human harm caused by dust, and greatly improve the quality of sucrose fatty acid esters.

I think the two key points of this process are **cooling and freezing**. The spray cooling conditions of the atomizer are as follows: the speed of the atomizer is 6000~22000rpm, the inlet temperature is 15 °C 20 °C, the outlet temperature is 35 °C 45 °C, the feed temperature is 55 °C 95 °C, and the feed speed is 30~100L/h. The freezing process in the cooling tower was as follows: granular sucrose fatty acid esters were kept at-40 °C for 4 hours, then heated to-28 °C for 30 hours within 1.2 hours, then heated to-12 °C for 10 °C and maintained for 5 hours within 4 hours, and then heated to 20 °C for 7 hours, the pressure of the whole freezing process was maintained at 0.01~100pa. The freezing process in the cooling tower is as follows: granular sucrose fatty acid esters

are maintained at -45 °C for 5 h, then heated to -25 °C within 1.5 h and maintained for 30 h, then heated to -10 °C within 5 h and maintained for 5 h, then heated to 20 °C and maintained for 7 h.

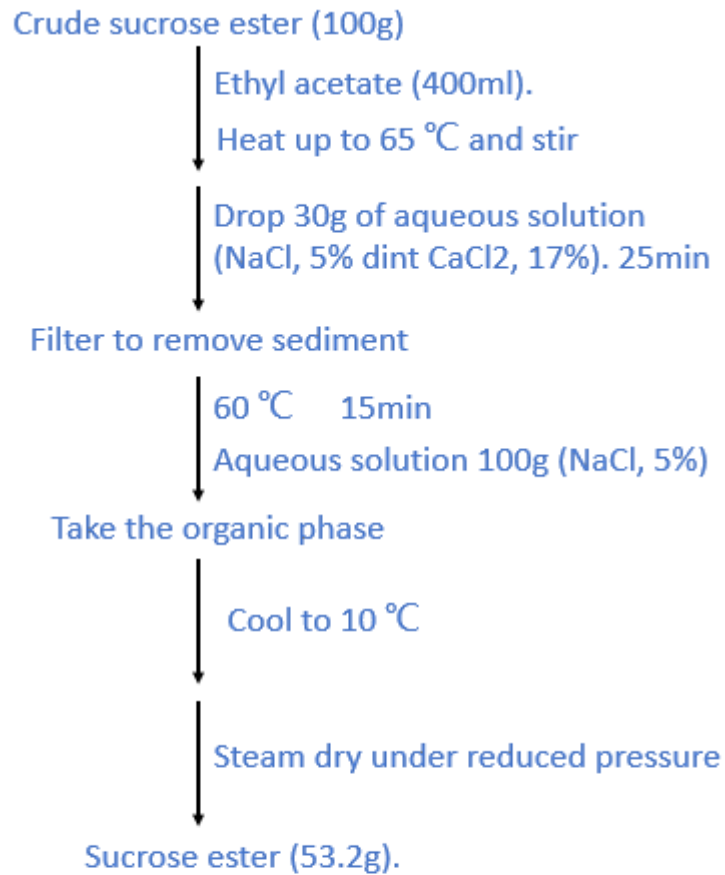


Gaotong03 A method for purification of sucrose ester

(CN 103087118 A)

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 200510006011.1 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 °C-40 °C 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%.



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%.