Research Report

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20240409

In the past two days, I have looked up some patents on solvent-free synthesis of sucrose esters, and studied and reviewed them. In the next two days (before next Monday), I will consult and study the patents of Gaotong related equipment.

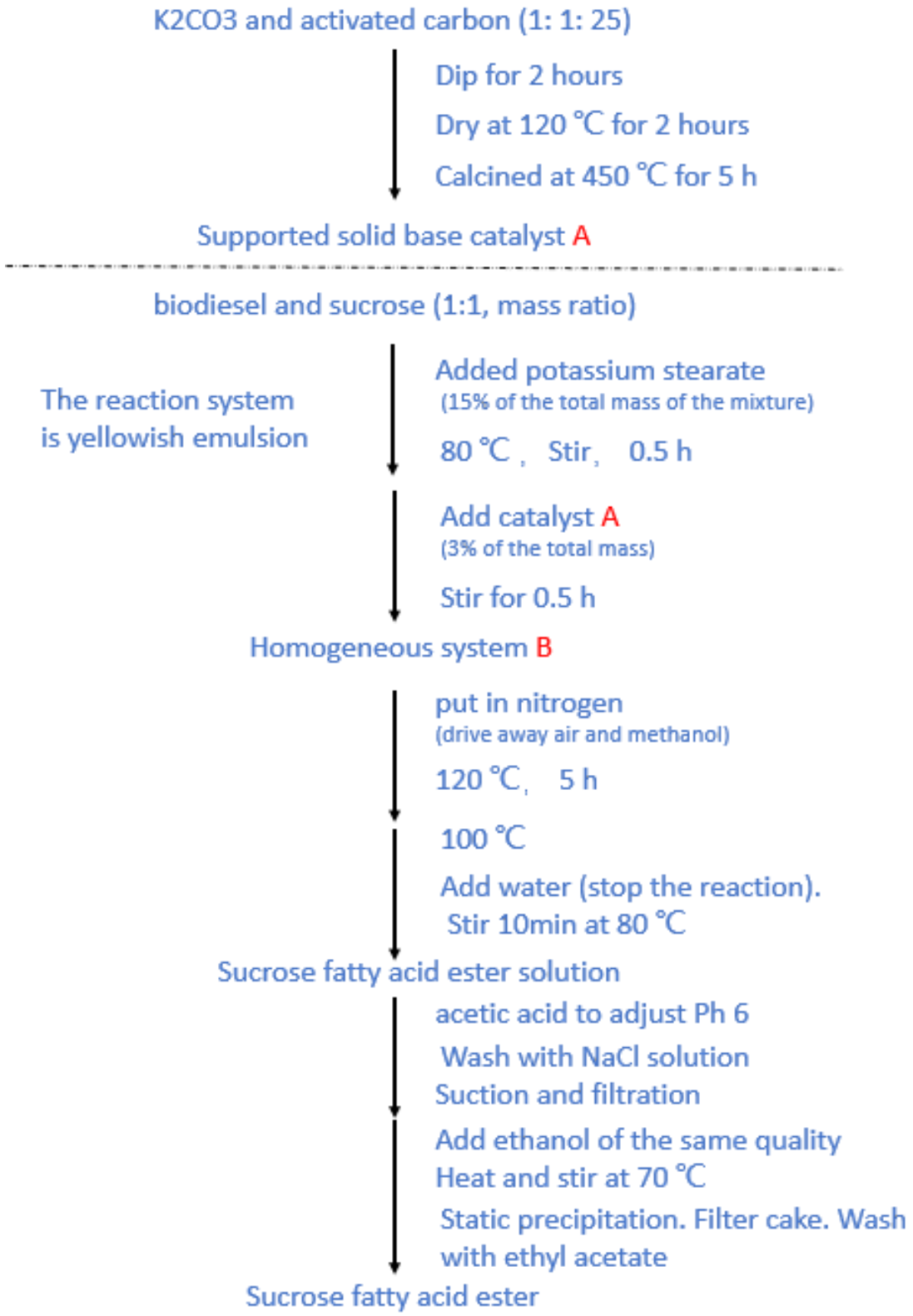
A method for direct synthesis of sucrose fatty acid esters from biodiesel and sucrose

(CN 101805381B)

The study on the synthesis of sucrose ester by solvent-free method has been widely carried out at home and abroad. Sun Shudong pointed out in the study on solvent-free synthesis of sucrose ester that cottonseed oil ethyl ester was prepared by the reaction of cottonseed oil with ethanol under the action of alkaline catalyst. then using sucrose and cottonseed oil ethyl ester as raw materials, sucrose: fatty acid ethyl ester = 1: 0.8, soap dosage 15%, catalyst amount: 2%, reaction temperature 135 ℃ Sucrose ester was synthesized by controlling reaction pressure ≤ 666Pa and reaction time 3 h. The crude product was first extracted with the aqueous solution of ethanol and sodium chloride, and then acetic acid was added to adjust the pH value. After repeated operation, the crude product was washed with acetic acid and sodium chloride aqueous solution, and the refined product was obtained after vacuum filtration. The yield of the product was 80%. Hu Jianhua pointed out that **sucrose ester was synthesized from sucrose and methyl stearate by using sucrose and methyl stearate as raw materials, 15% soap, 3% catalyst, 3.5 h reaction and controlling vacuum 13kPa ~ 0.667kPa.** The crude product was neutralized, salted out, washed, dried, and finally soaked in ether for 24 hours to obtain a refined product with a yield of 82%. However, most of the solvent-free methods for the synthesis of sucrose fatty acid esters, including the above two methods, have the following shortcomings: The main results are as follows:

1. all react under vacuum conditions, the reaction conditions are harsh, the industrial feasibility is not great, and in the actual production, the system is unstable under vacuum and high temperature, and it is easy to produce foaming phenomenon, which leads to the loss of potassium stearate and easy coking of sucrose. The color of the product is dark, and the post-treatment is difficult, which increases the production cost. 2. the post-treatment process of the product is complex, the catalyst can not be recovered, the industrial production cost is high, and the amount of solvent is large, the environmental pollution is serious, and the industrial feasibility is not high. 3. The methanol produced in the reaction is difficult to recover in vacuum, has high toxicity and serious environmental pollution.

This process is a method for direct synthesis of sucrose fatty acid esters from biodiesel and sucrose. Sucrose fatty acid ester was synthesized by solvent-free method under the protection of nitrogen (N2) at 1 atmospheric pressure, and the supported solid base catalyst was prepared by equal volume impregnation method. Then, after mixing biodiesel with sucrose, adding potassium stearate, reacting to the yellowish emulsion of the reaction system, adding the supported solid base catalyst, continuing the reaction, stirring and heating up, and adding nitrogen, driving away the air in the reaction system and taking away the methanol produced in the reaction process, heating up the reaction under the protection of N, cooling and stopping the flow of nitrogen, adding water to stop the reaction, and stirring 10min at 80 ℃. The refined product of sucrose fatty acid ester was obtained by treating the crude product. After the crude product is purified by solvent extraction, the purity of the product is more than 98%, the nitrogen can be reused, and the catalyst can be recycled.


K2CO3 and activated carbon (1: 1: 25)
Dip for 2 hours
Dry at 120 ℃ for 2 hours
Calcined at 450 ℃ for 5 h
Supported solid base catalyst A
biodiesel and sucrose (1:1, mass ratio)
The reaction system is yellowish emulsion
Added potassium stearate
(15% of the total mass of the mixture)
80 ℃， Stir， 0.5 h
Add catalyst A
(3% of the total mass)
Stir for 0.5 h
Homogeneous system B
put in nitrogen
(drive away air and methanol)
120 ℃， 5 h
100 ℃
Add water (stop the reaction).
Stir 10min at 80 ℃
Sucrose fatty acid ester solution
acetic acid to adjust Ph 6
Wash with NaCl solution
Suction and filtration
Add ethanol of the same quality
Heat and stir at 70 ℃
Static precipitation. Filter cake. Wash with ethyl acetate
Sucrose fatty acid ester

Raw material formula and synthesis method for synthesizing sucrose fatty acid

(CN 101514218 B)

In the process of synthesizing sucrose esters, in order to solve the insolubility of sucrose, fatty acids and their derivatives, modern industry mostly uses strong polar solvents to synthesize sucrose esters. The solvent method has the advantages of simple process, mild reaction conditions and high yield of sucrose ester. However, the reaction cost of the solvent method is expensive, and the residual solvent is difficult to remove, and the product is difficult to meet the standard of medical consumption. The solvent-free method can avoid the use of organic reagents and solve the toxicity problem, but the sucrose ester produced by the solvent-free method generally has low yield, low monoester content and dark color. Therefore, how to develop a method for the synthesis of sucrose esters without solvent, high monoester content and good color is an urgent problem for technicians in this field.

at present, transesterification is mostly used in the industrial synthesis of sucrose esters. Industrial transesterification synthesis of sucrose esters can be divided into two categories: solvent method and solvent-free method. Although the synthesis of sucrose ester by solvent-free method is non-toxic, short time and low cost, it is difficult to achieve eutectic of sucrose and fatty acid methyl ester (or ethyl ester) in the reaction system under the action of single fatty acid soap, resulting in uneven reaction system and difficult to reach homogeneous level. as a result, the content of sucrose monoester and total sucrose ester in the reaction product is not high, and the conversion rate is low. The sucrose ester products with high monoester content are widely used in food, medicine and many other fields, and the market demand is the greatest, so the promotion and application of solvent-free synthesis is greatly limited.

The formula of the process is as follows: sucrose: 50Mel 200 parts by weight, fatty acid methyl ester or ethyl ester: 20 parts by weight, catalyst: 0.2 parts by weight, fatty acid cationic soap salt: 5 parts by weight, modified phospholipids: 1-20 parts by weight, sucrose ester (HL.B 5-15): 0-8 parts by weight, sodium lactate: 6 parts by weight. In this process, the compound catalyst was added to the system of sucrose ester synthesis by solvent-free method. Among them, the addition of modified phospholipids can make the reaction of sucrose ester synthesis system more uniform and reach the eutectic state quickly, so as to effectively improve the conversion of sucrose ester and the content of sucrose monoester and total ester in the product.

1000ml three-neck bottle.
Fatty acid methyl ester (35g).

↓ 80-100 ℃. Stir.
Accelerator (HLB11) 3 grams
potassium stearate 30 grams

↓ magnesium carbonate 1.5 grams
calcium oxide2 grams
stirring well

↓ sucrose powder 160 grams
140-142 ℃， Vacuum 1500Pa
Stir and react for 65 minutes

Crude sucrose ester products

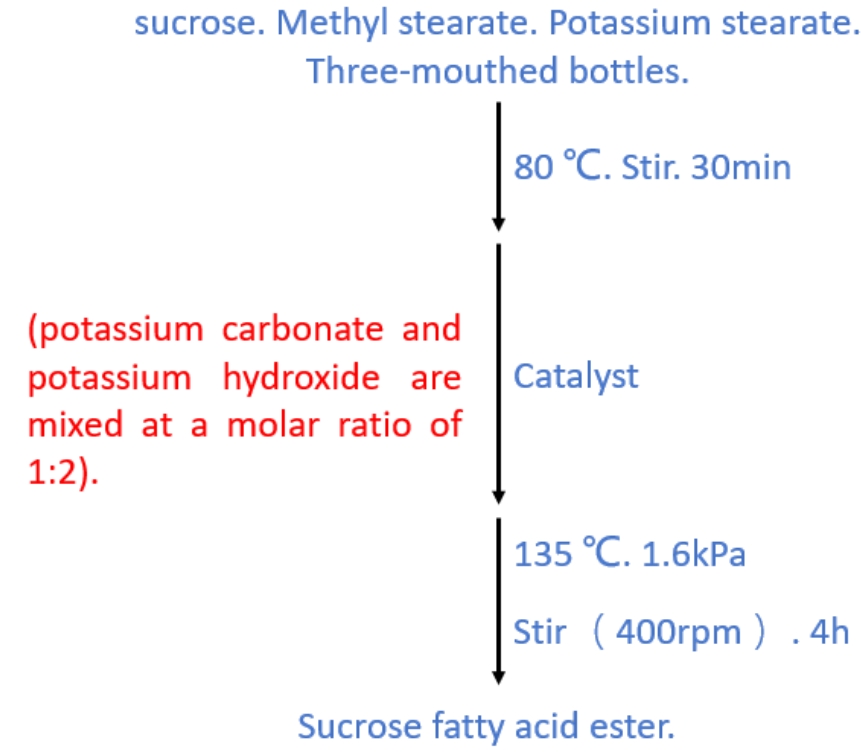
The yield is 69.32%. The content of sucrose monoester is 57.82%. The total ester content of sucrose is 95.76%.

A solvent-free method for synthesis of sucrose esters with high monoester content

(CN 114929720 B)

In the process of synthesizing sucrose esters, in order to solve the insolubility of sucrose, fatty acids and their derivatives, modern industry mostly uses strong polar solvents to synthesize sucrose esters. The solvent method has the advantages of simple process, mild reaction conditions and high yield of sucrose ester. However, the reaction cost of the solvent method is expensive, and the residual solvent is difficult to remove, and the product is difficult to meet the standard of medical consumption. The solvent-free method can avoid the use of organic reagents and solve the toxicity problem, but the sucrose ester produced by the solvent-free method generally has low yield, low monoester content and dark color. Therefore, how to develop a method for the synthesis of sucrose esters without solvent, high monoester content and good color is an urgent problem for technicians in this field.

The process is a solvent-free synthesis method of sucrose ester with high monoester content. The method comprises the following steps: (1) sucrose, higher fatty acid ester and higher fatty acid salt are mixed and preheated at 70-90 ℃ to obtain the mixture; (2) the mixture is mixed with the catalyst and reacted at 120-140 ℃ to obtain the sucrose ester. The synthesis method involved in this application adopts the solvent-free transesterification method, in which sucrose is co-melted with higher fatty acid esters, higher fatty acid esters and catalysts at a certain proportion at high temperature, and under reaction conditions conducive to kinetics, sucrose ester is synthesized by catalysis. The synthesis method avoids the use of solvents, solves the toxicity problem, makes the product reach the standard of medical consumption, the content of sucrose monoester in the product is high, the color of the product is good, the synthesis method is simple, and it is convenient for industrial production. It has important application value.



the molar ratio of sucrose to methyl stearate is 1purl; the mass of potassium stearate is 10% of the total mass of sucrose and methyl stearate; and the molar ratio of potassium ion to sucrose in the catalyst is 2:5.