Research Report

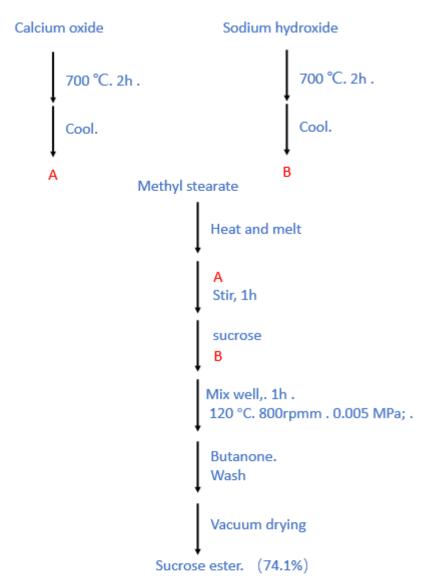
(Yufeng Hui)

20240419

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 patents of other companys.

A preparation method of sucrose fatty acid ester with low ash content (CN 106243166 B)

Sucrose fatty acid ester is referred to as sucrose ester (SE), which takes the hydroxyl part of sucrose as hydrophilic group and the carbon chain part of fatty acid as hydrophobic group. It is a kind of Nonionic green surfactant. Sucrose fatty acid esters, which are produced from natural renewable resources, are widely used in food, medicine, cosmetics and other industries because of their safety, non-toxic, non-irritating, non-pollution, biodegradable and other characteristics. The earliest synthesis of sucrose esters can be traced back to the 19th century. Sucrose was first esterified directly with fatty acid esters in the presence of toxic solvents. Because the esterification reaction is reversible, the reaction process is generally slow and the side reaction is complex. In order to enhance the positive charge of carbon-based carbon, Kea et al proposed to use fatty acid acyl chloride instead of fatty acid to obtain sucrose ester with high monoester content, but acyl chloride is toxic, which limits the wide application of sucrose ester (US:4683299). So people put forward the transesterification method, which dissolves sucrose and fatty acid esters into a phase with DMSO or DMF as solvent, and then carries on the transesterification reaction. However, the solvent method brings the problem of solvent residue, which can not meet the standards of food, cosmetics, pharmaceuticals and other industries. Sucrose is a combination of glucose and fructose. When heated to a certain temperature, sucrose is easily dehydrated. In the synthetic system of sucrose ester, sucrose is easily decomposed into glucose and fructose in the presence of water, and the thermal stability of glucose and fructose is lower than that of sucrose. Caramelization reaction is easy to occur at lower temperature, resulting in coking of the product and affecting the quality of the product. The process is a method for preparing sucrose ester, which comprises the following steps: (1) burning alkaline earth metal oxides and catalysts respectively and cooling them aside; (2) heating and melting fatty acid methyl esters, adding alkaline earth metal oxides after burning, stirring and filtering; (3) step (2) putting filtered fatty acid methyl esters, sucrose and burned catalysts in a reaction container, fully mixing and stirring the reaction in an oil bath. (4) the product obtained in step (3) is washed with solvent for many times, and the sucrose ester is obtained after vacuum drying. The process is simple and easy, the reaction is uniform, the conversion rate is high, and the obtained sucrose ester product has good color, high monoester content and good emulsifying performance, and is suitable for industrial production.



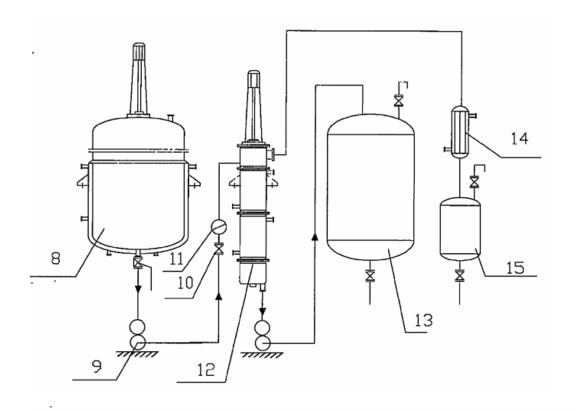
The dosage of the sodium hydroxide is 4% of the mass of the methyl stearate; the molar ratio of the methyl stearate to sucrose is 3Rom 1; and the amount of calcium oxide is 10% of the mass of the methyl stearate.

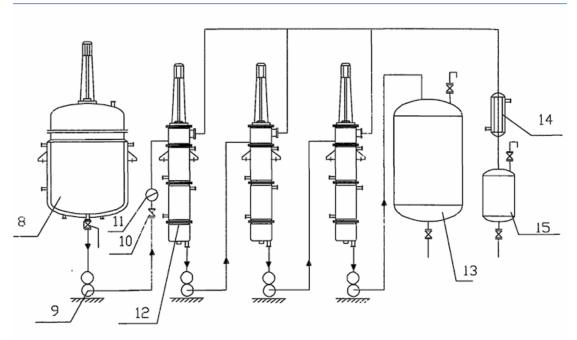
Membrane continuous transesterification reactor

(CN 201912933 U)

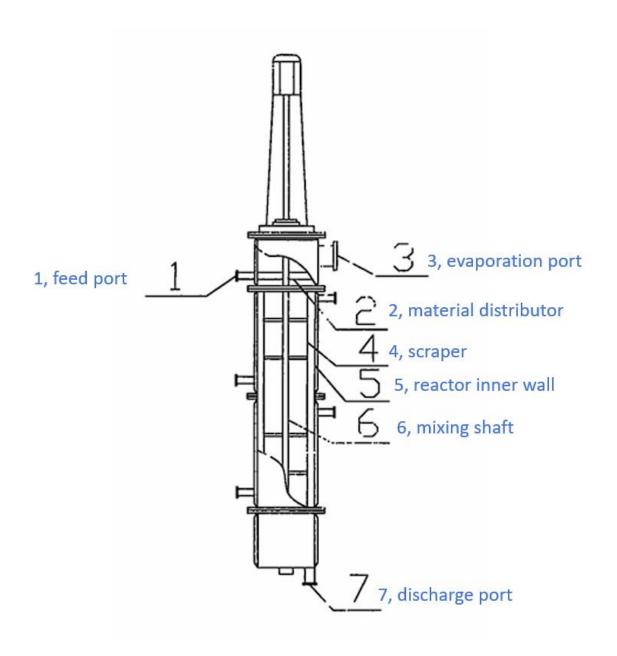
In organic chemical synthesis, transesterification is the reaction of ester and alcohol to form a new ester and a new alcohol under the catalysis of acid or base, that is, the alcoholysis of ester. The reaction is reversible and there is a reaction equilibrium. In order to make the reaction proceed in the required direction, the product needs to be removed from the reaction system. For example, the ester or alcohol with lower boiling point can be evaporated by distillation. Esters and acids can also undergo transesterification under certain conditions to form a new ester and a new acid, and it is also necessary to steam out the ester or acid with lower boiling point. The method of evaporating the product out of the reaction system is usually carried out in the reactor with distillation unit. This method belongs to intermittent production, the production cycle is long, and the conversion rate of raw materials is not ideal, which is not conducive to large-scale production. Production efficiency and quality are affected.

The device relates to a continuous transesterification reactor. The structure of the continuous rotating film transesterification reactor is as follows: the discharge end of the raw material mixing kettle is connected with the feed port of the rotating thin film reactor. The discharge port of the rotating thin film reactor is connected with the product storage tank. The evaporation port of the rotating thin film reactor is connected with the condenser. The condenser is connected to the receiver. When the material carries out the transesterification reaction in this reactor, the low boiling point substance produced is quickly removed from the reaction system, which is beneficial to the reaction in the direction of the product, and the reaction speed and reaction quality are greatly improved. because the material is flowing in the device, it can be continuously fed into production, which is conducive to large-scale production and improve production efficiency.





1, feed port, 2, material distributor, 3, evaporation port, 4, scraper, 5, reactor inner wall, 6, mixing shaft, 7, discharge port, 8, raw material mixing kettle, 9, gear pump, 10, material control valve, 11, Flowmeter, 12, rotating film reactor, 13, product storage tank, 14, condenser, 15, receiver.

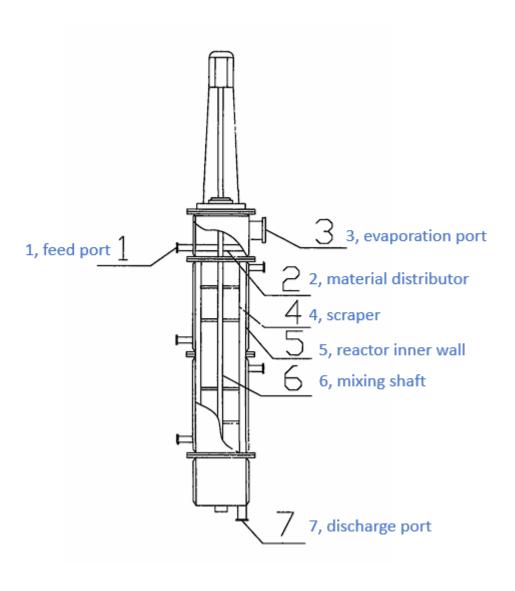


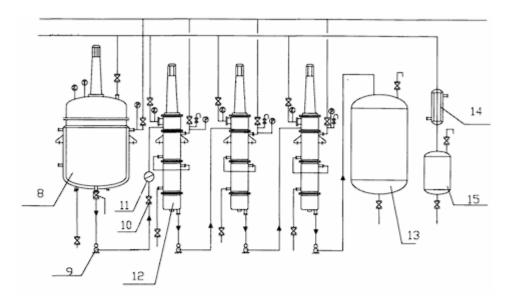
Synthesis of Sucrose Polyester by rotating Film transesterification continuous reactor

(CN 201912933 U)

In organic chemical synthesis, transesterification is the reaction of ester and alcohol to form a new ester and a new alcohol under the catalysis of acid or base, that is, the alcoholics of ester. The reaction is reversible and there is a reaction equilibrium. In order to make the reaction proceed in the required direction, the product needs to be removed from the reaction system. For example, the ester or alcohol with lower boiling point can be evaporated by distillation. Esters and acids can also undergo transesterification under certain conditions to form a new ester and a new acid, and it is also necessary to steam out the ester or acid with lower boiling point. The existing process for the synthesis of sucrose polyesters is a method of evaporating the products out of the reaction system, which is carried out in a reactor with a distillation unit. This method belongs to intermittent production, the production cycle is long, and the conversion rate of raw materials is not ideal. It is not conducive to large-scale production, affecting production efficiency and quality. In addition, the existing process for the synthesis of sucrose polyesters has the advantages of low yield, difficult separation and recovery of extraction residue in the extraction process, complex process, high production cost and low resource utilization.

The invention discloses a process for synthesizing sucrose polyesters, in particular to a process for synthesizing sucrose polyesters by using a rotating film transesterification continuous reactor. Methyl oleate, sucrose powder, potassium stearate and potassium carbonate were added into the mixing kettle and heated under stirring, and the material went into the rotating film transesterification continuous reactor for reaction, the crude fatty acid sucrose polyesters obtained after the reaction entered the product storage tank. Methanol, the product obtained after the reaction, was cooled by the condenser and entered the methanol receiver, and the crude product in the product tank was neutralized to neutral with acetic acid. Sucrose polyesters were obtained after washing and drying. The low boiling point substance generated by the process of the invention is quickly removed from the reaction system, which is beneficial to the reaction in the direction of the product, greatly improves the reaction speed and product quality, can be continuously fed into production, is conducive to large-scale production, and improves production efficiency. And the yield is high, the process is simple, the production cost is reduced and the resource utilization rate is improved.





1, feed port, 2, material distributor, 3, evaporation port, 4, scraper, 5, reactor inner wall, 6, mixing shaft, 7, discharge port, 8, raw material mixing kettle, 9, gear pump, 10, material control valve, 11, Flowmeter, 12, rotating film reactor, 13, product storage tank, 14, condenser, 15, methanol receiver.