(19) State Intellectual Property Office of the People's Republic of China

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(12) Invention patent

(10) Announcement No. CN 103360434 B

(45) Award tB 2016.05.04

(21) Application No. 2013103218403

(22) i please B 2013. 07.29

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45102

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(51) Int. CI.

"Porous metal H 13/06(2006/01)"

") ", "Breakbone H 1/00(2006.01)

"(56) to E file

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权利要求书1页 说明书4页

(54) Name of invention

Method for preparing sucrose ester

(57) Abstract

The invention discloses a method for preparing sucrose ester, which comprises the following steps :(1) alkali earth metal oxide and catalyst are respectively burned, cooled and reserved; (2) the fatty acid methyl ester is heated and melted, and the burned alkali earth metal oxide is added to stir and filter; (3) Put the filtered fatty acid methyl ester, sucrose and the burned catalyst in the reaction vessel after step (2), mix well, stir the reaction in the oil bath; (4) the product obtained in step (3) is washed several times with solvent, and the sucrose ester is obtained after vacuum drying. The invention has the advantages of simple and easy operation, uniform reaction, high conversion rate, good color and color of the obtained sucrose ester product, high single ester content and good emulsification performance, and is suitable for industrial production.

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1. A method for preparing sucrose ester is characterized in including the following steps:

(1) Calcium oxide and catalyst are respectively burned at 700℃~900℃ for 2~4 hours, then cooled for use;

(2) Heat the methyl stearate to melt, add the calcium oxide after burning in step (1), stir, and filter after stirring;

(3) the methyl stearate after step (2) filtration, sucrose and the catalyst after step (1) burning is placed in the reaction vessel, fully mixed, stirring in the oil bath for 1 to 3 hours, the temperature during the reaction is 120° c ~150°c, the stirring speed is 800 rpm~1000 RPM, the pressure is 0.005 MPa to 0.020 MPa;

(4) the product obtained from step (3) is washed several times with solvent, and the sucrose ester is obtained after vacuum drying;

The catalyst is one of sodium hydroxide and potassium hydroxide, and the amount of the catalyst is 4% to 8% of the quality of methyl stearate; The molar ratio of the methyl stearate and sucrose is 3:1; The amount of calcium oxide is 10% to 14% of the quality of the methyl stearate; The solvent is a kind of butyl ketone and water.

The preparation method of sucrose ester

Technical field

[0001] The invention relates to the technical field of non-ionic surfactant, in particular to a method for preparing sucrose ester.

Background technology

[0002] Sucrose fatty acid ester, referred to as sucrose ester (SE), is a non-ionic green surfactant with the hydroxyl part of sucrose as a hydrophilic group and the carbon chain part of fatty acid as a hydrophobic group. As a natural renewable resources as raw materials production of sucrose fatty acid ester, because of safety, non-toxic, non-irritating, pollution-free, biodegradable and other characteristics, widely used in food, medicine, cosmetics and other industries.

[0003] The earliest synthesis of sucrose ester dates back to the 19th century. Initially, sucrose was directly esterified with fatty acid esters in the presence of toxic solvents. Since esterification is a reversible reaction, the reaction process is generally slow and the side reactions are complex. In order to strengthen the electropositivity of base carbon,Kea et al proposed to replace fatty acid with acyl chloride, and obtained sucrose ester with high monoester content. However, acyl chloride was toxic, which limited the wide application of sucrose ester (US: 4683299). So people put forward the transesterification method, DMSO or DMF as the solvent, so that sucrose and fatty acid ester dissolved into a phase, and then transesterification reaction. However, the solvent method has brought about the problem of solvent residue, which can not meet the standards of food, cosmetics, pharmaceuticals and other industries.

[0004] Feuge et al proposed a solvent-free method for the synthesis of sucrose ester (Preparation of sucrose ester by intersterification, J Am Chen Soc, 1970), according to which sucrose must undergo a transesterification reaction with fatty acid esters in a molten state, so the reaction is carried out at a temperature of 170 to 190°C. At such high temperatures, sucrose is very unstable and easily coked, and the reaction usually has to be stopped in 2 to 20 minutes.

[0005] Sucrose is a combination of glucose and fructose. When heated to a certain temperature, sucrose tends to dehydrate. In the synthesis system of sucrose ester, if water is present, sucrose is easily decomposed into glucose and fructose. Glucose and fructose have lower thermal stability than sucrose, and are prone to caramelization at a lower temperature, causing the product to coking into lumps, affecting the quality of the product.

Content of invention

[0006] The invention aims to provide a method for preparing sucrose ester, which is carried out under strict anhydrous conditions, and can solve the problem that sucrose is easily decomposed into glucose and fructose in the presence of water, and glucose and fructose have lower thermal stability than sucrose, and are prone to caramelization at a lower temperature, resulting in the product coking into blocks and affecting the product quality.

[0007] In order to achieve the above purposes, the technical scheme adopted by the invention is: a method for preparing sucrose ester, which comprises the following steps:

[0008] (1) Alkali earth metal oxide and catalyst are respectively burned at 700℃~900℃ for 2~4 hours, cooled for use;

[0009] (2) The fatty acid methyl ester is heated to melt, adding the alkali earth metal oxide after the burning step (1), stirring, and filtering after stirring;

[0010] (3) the fatty acid methyl ester after step (2) filtration, sucrose and the catalyst after step (1) burning are placed in the reaction vessel, fully mixed, and stirred in the oil bath for 1 to 3 hours, the temperature during the reaction is 120℃~150° C, and the stirring speed is 800 rpm~1000rpm, the pressure is 0.005MPa ~0.020MPa;

[0011] (4) The product obtained from step (3) is washed several times with solvent, and the sucrose ester is obtained after vacuum drying;

[0012] the catalyst is one of sodium hydroxide and potassium hydroxide, and the amount of the catalyst is 4% to 8% of the quality of the fatty acid methyl ester; The fatty acid methyl ester is methyl stearate, the molar ratio of the fatty acid methyl ester and sucrose is 3:1; The alkaline earth metal oxide is calcium oxide, the alkali earth metal oxide dosage is 10% to 14% of the quality of the fatty acid methyl ester; The solvent is a kind of butyl ketone and water.

[0013] By adopting the technical scheme, the invention has the following beneficial effects compared with the prior art:

[0014] 1. Sucrose ester is obtained by transesterification reaction between sucrose and fatty acid methyl ester under strict anhydrous conditions, which can effectively prevent the occurrence of caramelization reaction, and has the characteristics of uniform reaction, high conversion rate and good product quality.

[0015] 2, the operation is simple and easy, the reaction is uniform, and the yield is higher than that of the general solvent-free synthesis, the single ester content is high, the emulsification performance is good, the environment is friendly, the energy is reduced, the cost is reduced, and the industrial production is conducive.

Specific implementation mode

[0016] Further details of the invention are given below in conjunction with embodiments:

[0017] Embodiment 1

[0018] Calcination of calcium oxide and sodium hydroxide at 700 ° C for 2 hours, cooling and reserving; The methyl stearate is heated to melt, add the calcium oxide after the burning step (1), stir, stir and filter; The filtered methyl stearate, sucrose and the sodium hydroxide after step (2) are placed in the reaction container, fully mixed, and stirred in the oil bath for 1 hour, the temperature during the reaction process is 120° C, the stirring speed is 800rpmm, and the pressure is 0.005MPa; The product obtained in step (3) is washed with butyl ketone for several times, and the sucrose ester is obtained after vacuum drying, with a yield of 74.1%.

[0019] The amount of sodium hydroxide is 4% of the mass of methyl stearate; The molar ratio of the methyl stearate to sucrose is 3:1; The amount of calcium oxide is 10% of the mass of the methyl stearate.

[0020] Embodiment 2

[0021] Calcination of calcium oxide and sodium hydroxide at 800 ° C for 3 hours, cooling and reserving; The methyl stearate is heated to melt, add the calcium oxide after the burning step (1), stir, stir and filter; The filtered methyl stearate, sucrose and the sodium hydroxide after step (1) are placed in the reaction vessel, fully mixed, and stirred in the oil bath for 2 hours, the temperature during the reaction is 130° C, the stirring speed is 900rpmm, the pressure is 0.010MPa; The product obtained in step (3) is washed with butyl ketone for many times, and the sucrose ester is obtained after vacuum drying, with a yield of 75.4%.

[0022] The amount of sodium hydroxide is 6% of the mass of methyl stearate; The molar ratio of the methyl stearate to sucrose is 3:1; The amount of calcium oxide is 12% of the mass of the methyl stearate.

[0023] Embodiment 3

[0024] Calcium oxide and sodium hydroxide were respectively burned at 900℃ for 4 hours, then cooled for use; The methyl stearate is heated to melt, add the calcium oxide after the burning step (1), stir, stir and filter; Step (2) the filtered methyl stearate, sucrose and step (1) the burned sodium hydroxide are placed in the reaction container, fully mixed, stirred in the oil bath for 3 hours, the temperature during the reaction process is 150°C, the stirring speed is 1000rpm, the pressure is 0.020MPa; The product obtained in step (3) is washed with butyl ketone for several times, and the sucrose ester is obtained after vacuum drying, with a yield of 76.9%.

[0025] The amount of sodium hydroxide is 8% of the mass of methyl stearate; The molar ratio of the methyl stearate to sucrose is 3:1; The amount of calcium oxide is 14% of the mass of the methyl stearate.

[0026] Embodiment 4

[0027] Calcination of calcium oxide and potassium hydroxide at 700 ° C for 2 hours, cooling and reserving; The methyl stearate is heated to melt, add the calcium oxide after the burning step (1), stir, stir and filter; The methyl stearate and cane after filtration in step (2)

The sugar and the potassium hydroxide after the burning of step (1) are placed in the reaction container, fully mixed, and stirred in the oil bath for 1 hour, the temperature during the reaction is 120°C, the stirring speed is 800rpmm, and the pressure is 0.005MPa; The product obtained in step (3) is washed with butyl ketone for many times, and the sucrose ester is obtained after vacuum drying, with a yield of 75.2%.

[0028] The amount of potassium hydroxide is 4% of the mass of methyl stearate; The molar ratio of the methyl stearate to sucrose is 3:1; The amount of calcium oxide is 10% of the mass of the methyl stearate.

[0029] Embodiment 5

[0030] Calcium oxide and potassium hydroxide are respectively calcined at 800 ° C for 3 hours, cooled and set aside; The methyl stearate is heated to melt, add the calcium oxide after the burning step (1), stir, stir and filter; The filtered methyl stearate, sucrose and the potassium hydroxide after step (1) are placed in the reaction vessel, fully mixed, and stirred in the oil bath for 2 hours, the temperature during the reaction is 130°C, the stirring speed is 900rpmm, and the pressure is 0.010MPa; The product obtained in step (3) is washed with butyl ketone for several times, and the sucrose ester is obtained after vacuum drying, with a yield of 74.9%.

[0031] The amount of potassium hydroxide is 6% of the mass of methyl stearate; The molar ratio of the methyl stearate to sucrose is 3:1; The amount of calcium oxide is 12% of the mass of the methyl stearate.

[0032] Embodiment 6

[0033 Respectively calcinate calcium oxide and potassium hydroxide at 900°℃ for 4 hours, cool and set aside; heat and melt methyl stearate, add the calcium oxide after calcising in step (1), stir, and filter after stirring; Step (2) the filtered methyl stearate, sucrose and step (1) after the burning of the potassium hydroxide placed in the reaction vessel, fully mixed, stirring reaction in the oil bath for 3 hours, the temperature of the reaction process is 150°C, stirring speed is 1000rpmm, the pressure is 0.020 MPa; The product obtained in step (3) is washed with water for several times, and the sucrose ester is obtained after vacuum drying, with a yield of 76.5%.

[0034] The amount of potassium hydroxide is 8% of the mass of the fatty acid methyl ester; The molar ratio of the fatty acid methyl ester to sucrose is 3:1; The amount of calcium oxide is 14% of the mass of the fatty acid methyl ester.

[0035] For the sucrose ester product prepared in the above embodiment, the solubility of the product was investigated. The specific steps are as follows: take 6 test tubes, labeled 1, 2, 3, 4, 5, 6, respectively, add 4ml water, n-butanol, ethyl acetate, ethanol, n-butane, and then add equal weight sucrose ester respectively, and heat them in a 25C water bath to observe the dissolution. Then it was heated in the water bath at 40°C to observe the dissolution. The results are shown in Table 1. The product has high solubility in polar solvent; Poor solubility in non-polar solvents. According to the similarity and compatibility principle, the polarity of the sucrose ester synthesized is large, that is, the content of hydroxyl in the molecule is high, which indicates the high single ester rate of the product.

[0036] Table 1 Solubility of sucrose ester

[0037]

Solvent.	water.	N-butanol.	Ethyl acetate.	ethanol.	N-hexane
溶剂	水	正丁醇	乙酸乙酯	乙醇	正己烷
25℃	±	+	±	+	-
40℃	+	+	+	+	-

[0038] Soil: partially dissolved; +: easily soluble; : insoluble

[0039] For the sucrose ester product prepared in the above embodiment, we investigated its emulsifying properties. The specific steps are as follows: take 40ml prepared sucrose ester, mix 0.15% aqueous solution with distilled water and 40ml liquid paraffin in 100m1 stopper cylinder, shake it up and down for 5 times, park it for 1min, then shake it up and down for 5 times, then pour the emulsion into another stopper cylinder with stopwatch to record the emulsion score

The time required when the oil and water are 10ml. The sucrose ester prepared by the invention is measured by the above method and the required time is about 3.5~4.2min

[0040] The hydrophilic lipophilic balance value (HLB) of the sucrose ester product prepared in the above embodiment was determined. According to the principle of hydrophilic and lipophilic equilibrium value and the additive characteristics of hydrophilic and lipophilic equilibrium value, the HLB value of sucrose ester was obtained by using the relationship between non-ionic surfactants with known HLB value and water number (water number method). The specific steps are as follows: Tween80 and Span85 are used to prepare a mixture of various HLB values according to mass ratio (100%Tween80 has an HLB value of 15; 100% Spans85 has an HLB of 1.8; And 100% Tween40 has an HLB of 15.6). Weigh 0.5g of the mixture with various HLB values into a 50ml colorimetric tube and add 25ml of N, n-dimethylformamidobenzene mixture , shake dissolved, control temperature 25±1°c, the colorimetric tube is placed on the electromagnetic agitator, put a rotor in the tube, attached to a 3" font paper after the tube, start the agitator, by the burette slowly drop into the distilled water (because of the heat release when titration, it must slowly titration control constant temperature), titration to the end point from the side of the colorimetric tube when the 3" font is fuzzy, record the volume of distilled water consumed The number. Use a variety of different HLB values as the horizontal coordinate, the volume of distilled water consumed milliliters as the vertical coordinate, as a standard curve. Repeat the above steps, record the volume milliliters of distilled water consumed by sucrose ester products, and finally find the HLB value of sucrose ester products by the standard curve. The HLB value of the sucrose ester prepared by the invention is about :10.6~11.8. It indicates that the synthesized sucrose ester has strong hydrophilicity and low hydroxyl esterification degree on sucrose, that is, the content of single ester of sucrose ester is high, which is consistent with the above results. The product is suitable to be used as oil-in-water (0/W) emulsifier.

(19) State Intellectual Property Office of the People's Republic of China

(12) Utility model patent

(10) License Announcement No. CN 201912933 U (45) granted 2t B 2011.08.03

(21) Diameter 201020616739.2
(22) Please E 2010.11.22

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(51) Int. CI.

B01 By 19/18 (2006.01)

B01J 4/00 (2006. 01)

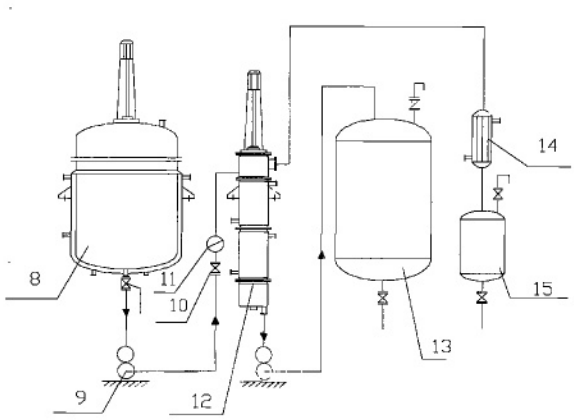
Claim 1 page description 3 pages with drawings 3 pages

(54) Use the new name

Rotary film transesterification continuous reactor

(57) Abstract

The utility model discloses a continuous reactor, in particular relates to a transesterification continuous reactor. The rotating film transesterification continuous reactor has the following structure: the discharge end of the raw material mixing kettle (8) is connected with the feed port (1) of the rotating film reactor (12), The discharge port (7) of the rotating film reactor (12) is connected with the product storage tank, the evaporation port (3) of the rotating film reactor (12) is connected with the condenser (14), and the condenser (14) is connected with the receiver (15). When the material is transesterification reaction in the reactor of the utility model, the generated low boiling point substance is quickly removed from the reaction system, which is conducive to the reaction in the direction of the product, greatly improving the reaction speed and reaction quality. Because the material is flowing in the device, it can be continuously fed into the production, which is conducive to large-scale production and improve the production efficiency.



1. A rotating film transesterification continuous reactor characterized by the following structure: the discharge end of the raw material mixing kettle (8) is connected to the feed port (1) of the rotating film reactor (12), the discharge port (7) of the rotating film reactor (12) is connected to the product storage tank, the evaporation port (3) of the rotating film reactor (12) is connected to the condenser (14), and the condenser (14) is connected to the feed port of the rotating film reactor (12). 14) is connected with the receiver (15).

2. According to the rotating film transesternization continuous reactor described in claim 1, the structure of the rotating film reactor (12) is as follows: the simple body is provided with a feed port (1) and an evaporation port (3), the upper part of the simple body is provided with a material distributor (2), the lower end of the simple body is provided with a discharge port (7), the inside of the simple body is provided with the inner wall of the reactor (5) and the scraper (4) Inside the inner wall of the reactor (5) and connected with the mixing shaft (6) in the middle of the barrel body.

3. The rotating film transesterification continuous reactor described in claim 1 or 2 is characterized in that the rotating film reactor (12) is one

4. According to the rotating film transesterification continuous reactor described in claim 1 or 2, it is characterized in that the rotating film reactor (12) is 2-8 units, each rotating film reactor (12) is connected in series, and the adjacent two rotating film reactors (12) are connected through the gear pump.

5, according to the rotating film transesterification continuous reactor described in claim 1, which is characterized in that the discharge end of the raw material mixing kettle (8) is connected with the feed port of the rotating film reactor (12) through the gear pump (9) and the pipeline.

6, according to the rotating film transesterification continuous reactor described in claim 5, which is characterized in that the pipeline is provided with a material regulating valve (10) and a flowmeter (11).

The rotating film transesterification continuous reactor

Technical areas

[0001] The utility model relates to a continuous reactor, in particular to a continuous transesterification reactor, which is suitable for the continuous production of biodiesel, sucrose polyester, sucrose fatty acid ester and other products requiring transesterification reaction.

Background technology

[0002] In organic chemical synthesis reaction, transesterification reaction is the reaction of ester and alcohol under the catalysis of acid or base to form a new ester and a new alcohol, that is, the alcoholysis reaction of ester. This reaction is reversible, there is a reaction equilibrium, in order for the reaction to proceed in the desired direction, the product needs to be removed from the reaction system. For example, the resulting esters or alcohols with lower boiling points can be steamed out by distillation. Ester and acid can also be transesterification reaction under certain conditions, to produce a new ester and a new acid, also need to produce a lower boiling point of the ester or acid to steam out.

[0003] The method of steaming the product out of the reaction system is usually carried out in the reaction kettle installed with a distillation device. This method belongs to intermittent production, has a long production cycle, and the conversion rate of raw materials is not ideal, which is not conducive to large-scale production and affects the production efficiency and quality.

Content of utility model

[0004] In order to solve the above technical problems, the utility model proposes a rotating film transesterification continuous reactor. When the material is transesterification reaction in the reactor of the utility model, the generated low boiling point substance is rapidly removed from the reaction system, which is conducive to the reaction in the direction of the product, greatly improving the reaction speed and reaction quality, because the material is flowing in the device and can be continuous The sexual feeding production is conducive to large-scale production and improve production efficiency.

[0005] In order to solve the above technical problems, the utility model is realized through the following technical scheme:

[0006] A rotating film transesterification continuous reactor has the following structure: the discharge end of the raw material mixing kettle is connected with the feed port of the rotating film reactor, the discharge port of the rotating film reactor is connected with the product storage tank, the evaporation port of the rotating film reactor is connected with the condenser, and the condenser is connected with the receiver.

[0007] The structure of the rotating film reactor is as follows: the simple body is provided with a feed port and an evaporation port, the upper part of the simple body is provided with a material distributor, the lower end of the simple body is provided with a discharge port, the simple body is provided with the inner wall of the reactor, the scraper is arranged on the inner wall of the reactor and connected with the mixing shaft in the middle of the simple body.

[00083 the rotating film reactor of the row is 1.

[000☒ the rotating film reactor is 2-8 sets, and each rotating film reactor is connected in series, and the adjacent two rotating film reactors are connected through a gear pump.

[0010] The discharge end of the raw material mixing kettle is connected with the feed port of the rotating film reactor through the gear pump and the pipeline.

[0011] The pipeline is provided with a material regulating valve and a flowmeter.

[0012] Because of the structure, the advantages and effects of the utility model are as follows

[0013] When the material is transesterification reaction in the reactor of the utility model, the generated low boiling point substance is quickly removed from the reaction system, which is conducive to the reaction in the direction of the product, and greatly improves the reaction speed and reaction quality. Because the material is flowing in the device, it can be continuously fed for production, which is conducive to large-scale production and improves the production efficiency.

The accompanying diagram illustrates

[0014] FIG. 1 is a schematic diagram of the structure of a single-stage rotating film transesterification continuous reactor.

[0015] FIG. 2 shows the structure diagram of the three-stage rotating film transesterification continuous reactor.

[0016] Figure 3 is a schematic of the structure of the rotating film reactor in Figures 1 and 2.

[0017 In the figure,1, feed port,2, material distributor,3, evaporation port,4, scraper,5, reactor wall,6, mixing shaft,7, out Feed port,8, raw material mixing kettle,9, gear pump,10, material regulating valve,11, flow meter, 12, rotating film reactor,13, product Storage tank,14, condenser, 15, receiver.

Specific implementation method

[0018] The utility model is further explained in combination with the attached drawings.

[0019] Embodiment 1

[0020] As shown in Figure 1 and Figure 3, the structure of the rotary film transesternization continuous reactor of the utility model is as follows: the discharge end of the raw material mixing kettle 8 is connected with the feed port of the rotating film reactor 12 through the gear pump 9 and a pipeline, the pipeline is provided with a material regulating valve 10 and a flow meter 11, the discharge port 7 of the rotating film reactor 12 is connected with the product storage tank 13, and the rotating film reactor 12 is connected The evaporation port is connected with the condenser 14, and the condenser 14 is connected with the receiver 15. The structure of the rotating film reactor 12 is as follows: the simple body is provided with a feed port 1 and an evaporation port 3, the upper part of the simple body is provided with a material distributor 2, the lower end of the simple body is provided with a discharge port 7, the inside of the barrel is the inner wall of the reactor 5, and the scraper 4 is arranged on the inner wall of the reactor 5 and connected with the mixing shaft 6 in the middle of the barrel body.

[0021] Embodiment 2:

[0022] As shown in Figure 2 and Figure 3, the structure of the rotary film transesterification continuous reactor of the utility model is as follows:

[0023] The discharge end of the raw material mixing kettle 8 is connected with the feed port of the first rotary film reactor 12 through the gear pump 9 and the pipeline, the pipeline is provided with the material regulating valve 10 and the flow meter 11, the rotary film reactor 12 is three, the three rotary film reactor 12 is connected in series, the adjacent rotary film reactor is connected with the gear pump, and the third rotary film reactor is the discharge port Connected with the product storage tank 13, the evaporation port of the rotating film reactor 12 is connected with the condenser 14, and the condenser 14 is connected with the receiver 15. The structure of the rotating film reactor 12 is the same as that of Embodiment 1.

[0024] Embodiment 3:

[0025] The rotating film reactor 12 in embodiment 2 is two, the two rotating film reactors 12 are connected in series, the two rotating film reactors 12 are connected by a gear pump, and the other structures are the same as in embodiment 2

[0026] Embodiment 4:

[0027] The rotating film reactor 12 described in embodiment 2 is 4-8 units, each rotating film reactor 12 is connected in series, and two adjacent rotating film reactors 12 are connected by a gear pump. Other structures are the same as in embodiment 2.

[0028] The working principle of the utility model is as follows: the rotary film transesteric continuous reactor of the utility model is composed of a raw material mixing kettle, a rotating film reactor and a receiver, which is characterized by the use of a rotating film reactor to form a single stage or more rotating film reactors in series to form a multistage continuous reaction device, and the gear pump is connected between each rotating film reactor. The raw material mixing kettle mixed and preheated, has not yet reacted the material from the entrance tangentially into the rotary film reactor, the material distributor installed in the rotary film reactor barrel body is continuously and evenly distributed in the reactor wall, in the rotation of the scraper stirring action along the inner wall of the evaporator flow down, while forming a film, the process of the material transesternization reaction, the generation of low boiling point Product under vacuum conditions for falling film evaporation, quickly evaporated out of the reaction system, esterification reaction in the direction of the product, greatly improve the reaction speed, when the material reached the reactor outlet, has reached the end of the reaction, and low boiling point

The product has been effectively separated. This process is characterized by the unreacted material after the reactor reaction is no longer circulated to the mixing reactor, the logistics direction is one-way, conducive to continuous production, improve equipment production capacity and production efficiency, and the traditional batch esterification equipment is unreacted raw materials and products mixed in the same system, there is a reaction balance, it takes a long time to reach the reaction end point, because Is the intermittent production, the output is limited, the production efficiency is not high.

[0029] The material for transesterification reaction of the utility model is reacted in a rotating reactor and forced to form a film through a rotating scraper, and the generated product with low boiling point is evaporated in falling film under vacuum condition, and is quickly excluded from the reaction system. Because the film evaporator has large heat transfer coefficient, high evaporation intensity, short flow time and large operation elasticity, it is very suitable for use as a transesterification exchanger.

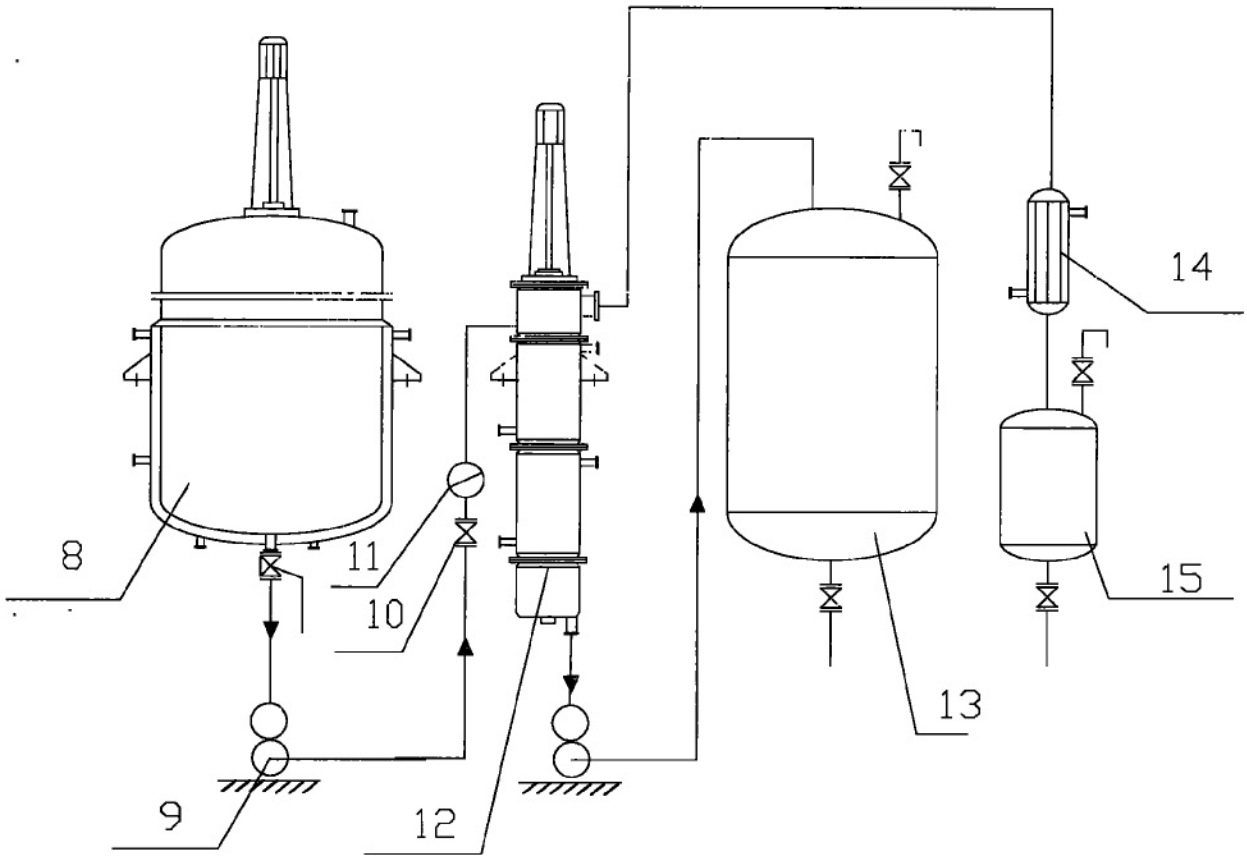


Figure 1

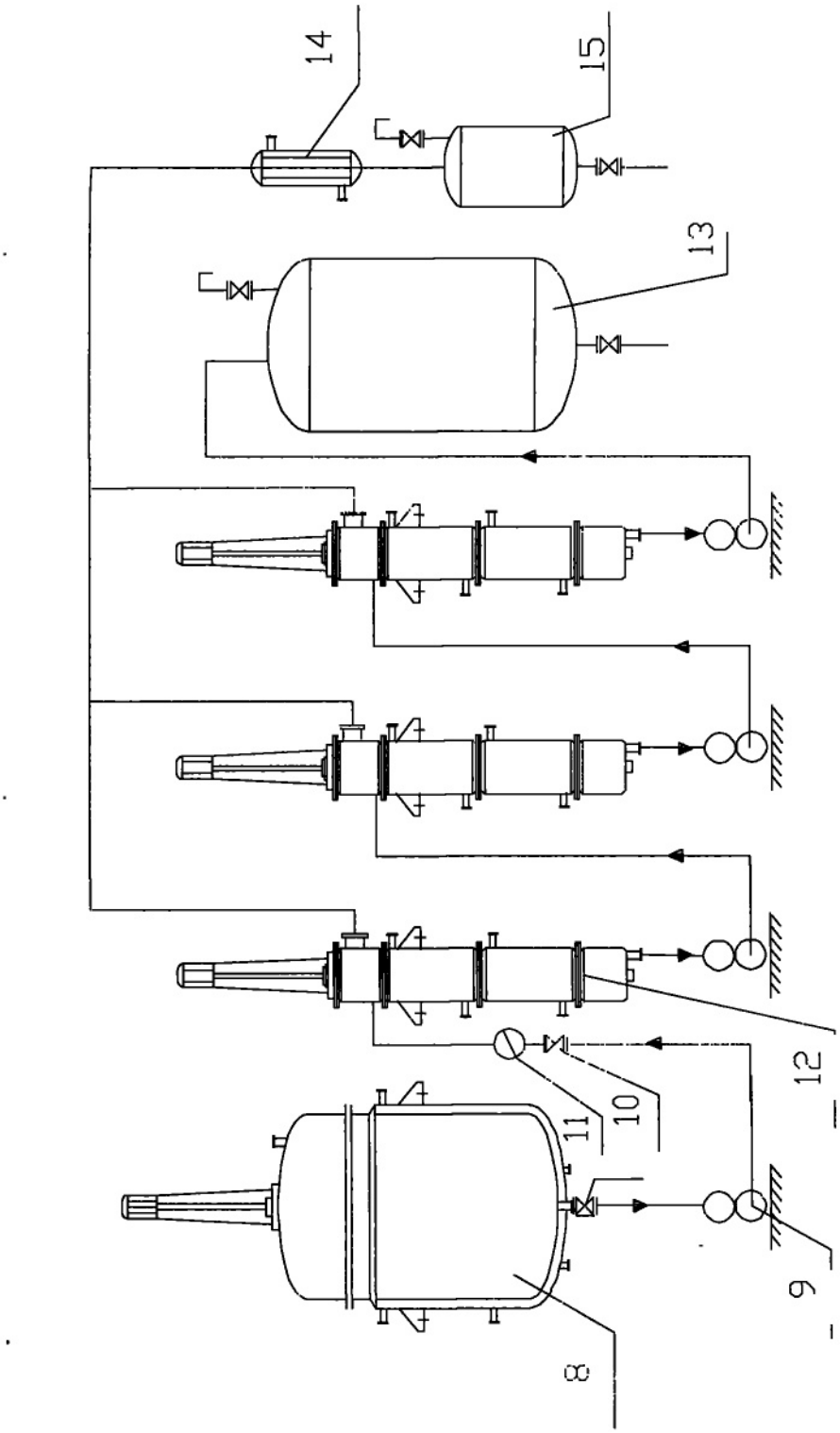


Figure 2

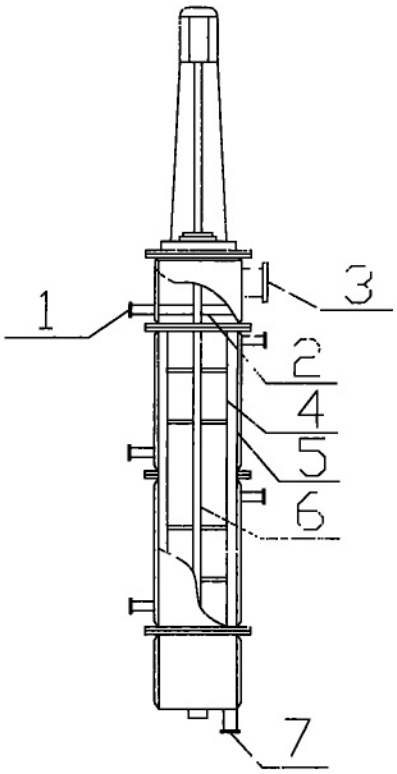


Figure 3

(19) State Intellectual Property Office of the People's Republic of China

(12) Application for invention patent

(10) Please refer to ☒ CN 102093438
(43) Please "ekB 2011.06.15"

It is (21) ϕ201010552399.6
(22) i please B 2010.11.22

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(51) Int. CI.

"Daresin 13/08 (2006.01)

"Darexas 1/00 (2006.01)

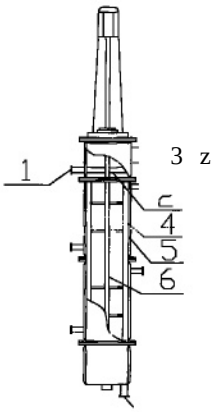
Claim 1 page description 3 pages with drawings 2 pages

(54) Name of invention

Process for synthesizing sucrose polyesters using a rotating film transesterification continuous reactor

(57) Abstract

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



1. A process for synthesizing sucrose polyesters using a rotating film transesterification continuous reactor, characterized by the addition of fatty acid methyl E in a mixing kettle 900-1100kg, sucrose powder 80-100kg, fatty acid potassium 25-35kg, potassium carbonate 25-35kg, stirred heating to 115℃, and in the whole process of production to maintain the material constant temperature, open the mixing kettle discharge valve, the material through the gear pump, into the rotating film transesterification continuous reactor for reaction, reaction temperature control 125-1 At 35℃, the reactor pressure is maintained at -0.1-0. 092MPa, the material flows through the reactor time takes about 10 minutes, the fatty acid sucrose polyester crude obtained after the reaction enters the product storage tank, the product methanol obtained after the reaction is cooled by the condenser into the methanol receiver, when the material in the mixing kettle and the reactor material flow empty, the reaction ends, the crude product in the product storage tank is neutralized to neutral with acetic acid, washed and dried Sucrose polyester products.

2. According to the process of synthesizing sucrose polyester using rotating film transesterification continuous reactor described in Claim 1, it is characterized by adding fatty acid methyl ester 1000kg into the mixing kettle. 200 objective sucrose powder 90kg, fatty acid potassium 30kg, potassium carbonate 30kg, stirred heating to 115℃, and in the whole process of production to maintain a constant temperature of the material, open the mixing kettle discharge valve, the material through the gear pump and the installation of steam sleeve material pipe, under the flow control valve regulation with 300L/h flow into the rotating film transesternization continuous reactor Reaction, reaction temperature control 125-135℃, reactor pressure maintained at -0.1-0. 092MPa, the material flows through the reactor time takes about 10 minutes, the fatty acid sucrose polyester crude obtained after the reaction enters the product storage tank, the product methanol obtained after the reaction is cooled by the condenser into the methanol receiver, when the material in the mixing kettle and the reactor material flow empty, the reaction ends, the crude product in the storage tank is neutralized to neutral with acetic acid, washing, drying after sucrose Polyester products.

3, according to the process of synthesizing sucrose polyester using rotating film transesterification continuous reactor described in claim 1 or 2, it is characterized in that the washing is washed under the temperature of 60℃ in turn with saturated salt water of 3 times the weight of crude product and ethanol of 3 times the weight of crude product.

4, according to claims 1 or 2 of the use of rotating film transesterification continuous reactor to synthesize sucrose polyester process, which is characterized by the structure of the rotating film transesterification continuous reactor is as follows: the simple body is provided with a feed port (1) and evaporation port (3), the upper part of the simple body is provided with a material distributor (2), the lower end of the simple body is provided with a discharge port (7), the simple body internal reaction The inner wall of the device (5) and the scraper (4) are arranged in the inner wall of the reactor (5) and connected with the mixing shaft (6) in the middle of the simple body.

The process of synthesizing sucrose polyester using rotating film transesterification continuous reactor

Technical field

[0001] The invention relates to a process for synthesizing sucrose polyester, in particular to a process for synthesizing sucrose polyester using a rotating film transesterification continuous reactor.

Background technology

[0002] In organic chemical synthesis reaction, transesterification reaction is the reaction of ester and alcohol under the catalysis of acid or base to form a new ester and a new alcohol, that is, the alcoholysis reaction of ester. This reaction is reversible, there is a reaction equilibrium, in order for the reaction to proceed in the desired direction, the product needs to be removed from the reaction system. For example, the resulting esters or alcohols with lower boiling points can be steamed out by distillation. Ester and acid can also be transesterification reaction under certain conditions, to produce a new ester and a new acid, also need to produce a lower boiling point of the ester or acid to steam out.

[0003] The existing process of synthesizing sucrose polyester is the method of steaming the product out of the reaction system, which is carried out in the reactor installed with a distillation device. This method belongs to intermittent production, long production cycle, raw material conversion rate is not ideal, which is not conducive to large-scale production, affecting the production efficiency and quality. In addition, the existing synthetic sucrose polyester process has low yield, difficult separation and recycling of extraction process raffinate, complicated process, high production cost and low resource utilization.

[0004] The invention patent (Application (patent) No. :CN03101111.X) of Xinjiang Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, "New process for synthesizing high esterification degree sucrose fatty acid polyester", relates to a new process for synthesizing high esterification degree sucrose fatty acid polyester, which is based on fatty acid methyl ester or fatty acid ethyl ester as raw material and is steamed repeatedly by thin film evaporator or molecular still Distillation, improve the efficiency of esterification reaction and esterification degree of product sucrose fatty acid polyester, although this method makes up for the low conversion rate of conventional batch production of raw materials, but the material is circulating at high temperature for a long time, the crude sucrose polyester is easy to color, in the purification process needs to use hydrogen peroxide and other methods of decolorization, resulting in high peroxide value of products, in some areas such as food machinery lubrication The application of health care products is limited; The process still belongs to batch production, and the production efficiency is not high in large-scale production.

Content of invention

[0005] In order to solve the above technical problems, the invention provides a process for synthesizing sucrose polyester using a rotating film transesterification continuous reactor, aiming at high reaction speed and reaction quality, realizing continuous feeding production, conducive to large-scale production, improving production efficiency, high yield, simple process, reducing production cost and improving resource utilization rate.

[0006] In order to solve the above technical problems, the invention is realized through the following technical solutions:

[0007] The invention provides a process of synthesizing sucrose polyester using a rotating film transesterification continuous reactor, adding fatty acid methyl ester 900-1100kg, sucrose powder 80-100kg, fatty acid potassium 25-35kg, potassium carbonate 25-35kg into the mixing kettle, heating to 115℃ under stirring, and maintaining constant temperature of materials in the whole process of production, opening the mixing kettle discharge valve, Material through the gear pump, into the rotating film transesterification continuous reactor for reaction, reaction temperature control 125-135℃, reactor pressure maintained at -0.1-0. 092MPa, the material flows through the reactor time takes about 10 minutes, the fatty acid sucrose polyester crude obtained after the reaction into the product storage tank, the product methanol obtained after the reaction is cooled by the condenser into the methanol receiver, when the material in the mixed kettle and the reactor of the material flow empty, the end of the reaction, the crude product in the product storage tank is neutralized to neutral with acetic acid, washing, drying Sucrose polyester products.

[0008] The process of synthesizing sucrose polyester using rotating film transesterification continuous reactor, adding fatty acid methyl ester 1000kg in the mixing kettle, 200 days of sucrose powder 90kg, fatty acid potassium 30kg, potassium carbonate 30kg, stirred heating to 115℃, and in the whole process of production to maintain a constant temperature of the material, open the mixing kettle discharge valve, the material through the gear pump and the installation of steam sleeve material pipe, under the flow control valve to 300L/h flow into the rotating film transesternization continuous reactor Reaction, reaction temperature control 125-135℃, reactor pressure maintained at -0. 1-0. 092MPa, the material flows through the reactor time takes about 10 minutes, the fatty acid sucrose polyester crude obtained after the reaction enters the product storage tank, the product methanol obtained after the reaction is cooled by the condenser into the methanol receiver, when the material in the mixing kettle and the reactor material flow empty, the reaction ends, the crude product in the storage tank is neutralized to neutral with acetic acid, washing, drying after sucrose Polyester products.

[0009] The washing is washed under the temperature of 60℃ with saturated salt water of 3 times the weight of crude product and ethanol of 3 times the weight of crude product in turn.

[0010] The structure of the rotating film transesterification continuous reactor is as follows: a simple body is provided with a feed port 1 and an evaporation port

3, the upper part of the simple body is provided with a material distributor 2, the lower end of the simple body is provided with a discharge port 7, the simple body is provided with the inner wall of the reactor 5, a scraper

4 is arranged in the inner wall of the reactor 5 inside, the middle of the simple body is provided with a stirring shaft 6.

[0011] The invention has the following advantages and effects due to the adoption of the process method:

[0012] The salt aqueous solution obtained by washing of the invention contains soap, potassium acetate and sucrose, which can be used to produce defoamer for paper. The ethanol solution contains fatty acids and a small amount of fatty acid methyl ester. After recovery, the fatty acids and fatty acid methyl ester are used to synthesize sucrose polyester, and ethanol is used to purify sucrose polyester.

[0013] The invention adopts a rotating film transesterification continuous reactor to shear the reactants into films in the molten homogeneous state by rotating high-speed stirring, which greatly increases the contact area between the reactants and the interface between the material and the vacuum, so that the by-product methanol generated by the reaction is easier to move out of the reaction system, so that the reaction is more conducive to the direction of generating sucrose polyester, and the appearance of the product is beautiful Good color is conducive to continuous production and improves equipment productivity and production efficiency.

[0014] The separation and recovery of extraction process raffinate solves the difficult problem of separation and recycling of extraction process raffinate, and has important significance for industrialization promotion, simplification of process, reduction of production cost and improvement of resource utilization rate.

[0015] The invention has the advantages of high yield, simple process, lower production cost and higher resource utilization rate.

Illustrated with drawings

[0016] FIG. 1 is a structural diagram of the rotary film transesterification continuous reactor of the invention.

[0017]

FIG. 2 is a process flow diagram of the invention.

[0018 In the figure,1, feed port,2, material distributor,3, evaporation port,4, scraper,5, reactor inner wall,6, stirring shaft,7, discharge port, 8, raw material mixing kettle,9, gear pump,10, material regulating valve,11, flow meter,12, rotating film reactor,13, product storage tank, 14, condenser, 15, methanol receiver.

Specific implementation mode

[0019] The invention is further explained in combination with embodiments below, but the scope of protection of the invention is not limited by embodiments.

[0020]

Example 1:

[0021] Add fatty acid methyl ester 1000kg to mixing kettle, 200 mesh sucrose powder 90kg, potassium fatty acid 30kg, potassium carbonate 30kg, heated to 115℃ under stirring, and maintain the material constant temperature during the whole process of production, open the mixing kettle discharge valve, the material through the gear pump and the material pipe installed with steam sleeve, under the flow control valve at 300L/h flow

Enter the rotating film transesterification continuous reactor for reaction, the reaction temperature is controlled 125-135℃, the reactor pressure is maintained at -0.1-0.092MPa, the material flows through the reactor time takes about 10 minutes, the fatty acid sucrose polyester crude obtained after the reaction enters the product storage tank, the product methanol obtained after the reaction is cooled by the condenser into the methanol receiver When the material in the mixed kettle and the reactor of the material flow empty, the end of the reaction, the crude products in the storage tank with acetic acid to neutral, at 60 ° C temperature in turn with 3 times the weight of the crude saturated salt water and 3 times the weight of the crude ethanol under the washing, after drying sucrose polyester products, product yield of 72% (relative fatty acid methyl ester).

[0022] The sucrose polyester obtained in this embodiment according to GB8272-2009 test results are: free sugar 0. 25%, acid value 2. 75mgKOH /g, ash 0.26%, esterification degree 7.1.

[0023] As shown in FIG. 1,

[0024] The structure of the rotating film transesternization continuous reactor mentioned in all the above embodiments is as follows: the simple body is provided with a feed port 1 and an evaporation port 3, the upper part of the simple body is provided with a material distributor 2, the lower end of the simple body is provided with a discharge port 7, the simple body is provided with an inner wall of the reactor 5, the scraper 4 is arranged on the inner wall of the reactor 5, the middle part of the simple body is provided with a stirring shaft 6, the invention rotates the film transesternization The continuous reactor can be 1, or it can be 2-8, if it is multiple, the rotating film reactor is connected in series, and the adjacent two rotating film reactors are connected through the gear pump.

[0025] The rotating film transesteric continuous reactor preferably adopted in the process of the invention is characterized in that a rotating film reactor is used to form a single stage or multiple rotating film reactors in series to form a multistage continuous reaction device, and each rotating film reactor is connected by a gear pump. The material mixed and preheated by the raw material mixing kettle, which has not yet reacted, enters the rotary film reactor tangentially from the entrance, and is continuously and evenly distributed on the inner wall of the reactor by the material distributor installed in the cylinder of the rotary film reactor, flows downward along the inner wall of the evaporator under the stirring of the rotating scraper, and forms a film at the same time. In this process, the material carries out the transesternization reaction and generates a low boiling point The product under vacuum conditions for falling film evaporation, quickly evaporated out of the reaction system, esterification reaction in the direction of the product, greatly improve the reaction speed, when the material reached the reactor outlet, has reached the end of the reaction, and the low boiling point of the product has been effectively separated. This process is characterized by the unreacted material after the reactor reaction is no longer circulated to the mixing reactor, the logistics direction is one-way, conducive to continuous production, improve equipment production capacity and production efficiency, and the traditional batch esterification equipment is unreacted raw materials and products mixed in the same system, there is a reaction balance, it takes a long time to reach the reaction end point, because Is the intermittent production, the output is limited, the production efficiency is not high.

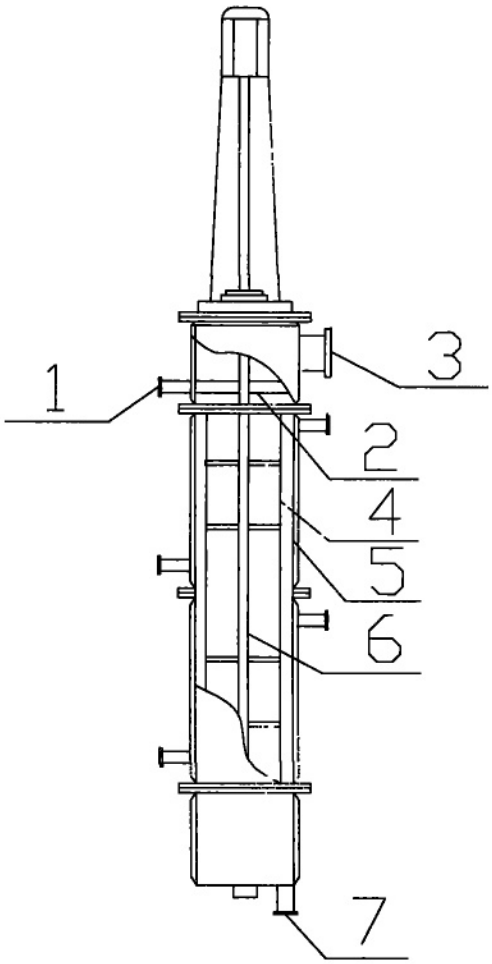


Figure 1

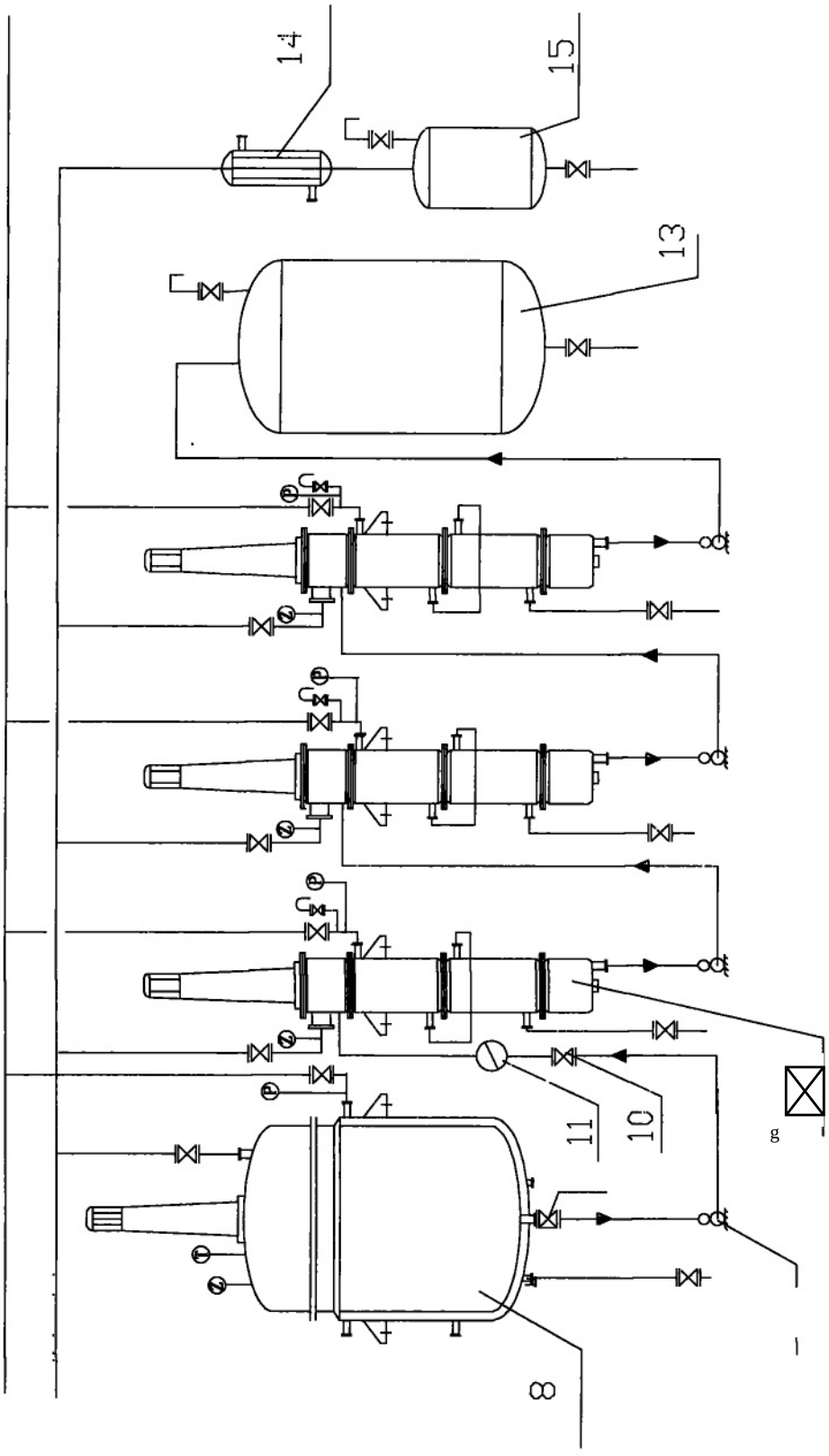


Figure 2