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(73) Patenthaver: **Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina**
Shanxi Lu'an Mining (Group) Co., Ltd., Houbao Town , Xiangyuan County, Changzhi, Shanxi, Kina

(72) Opfinder: **FAN, Weibin, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina**
WANG, Guofu, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
GUO, Jiaqi, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
WANG, Jianguo, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
DONG, Mei, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
CEN, Youliang, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
XIAO, Yaning, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
WANG, Dongfei, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
SUN, Shoujing, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
WANG, Weilin, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
ZHANG, Juncai, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
ZHANG, Min, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina
LI, Yunhong, Institute of Coal Chemistry, Chinese Academy of Sciences, 27 South Taoyuan Road, Taiyuan, Shanxi, Kina

(74) Fuldmægtig i Danmark: **Zacco Denmark A/S, Arne Jacobsens Allé 15, 2300 København S, Danmark**

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WO-A1-2012/011142
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DESCRIPTION

Description

TECHNICAL FIELD

[0001] The present invention belongs to the technical field of tank reactors, and specifically relates to a slurry-bed tank reactor configured for a use in which materials are continuously fed into and discharged from the tank reactor.

BACKGROUND

[0002] A tank reactor is widely applied in industry. On the basis of operation mode, the tank reactor may be classified as a batch tank reactor, a semibatch tank reactor and a continuous tank reactor. In the continuous tank reactor, materials are continuously fed and discharged to achieve continuous operations and high efficiency. However, it is still a challenge to continuously operate a heterogeneous reaction in a tank reactor. Chinese Patent Application No. 2016105989527 (published as CN106220481 A) provides a continuous operation method, but in the running process, solid powders are often adhered to tubular separation membranes, and hence, affecting the continuity of running, and the reflection of long catalytic life of a catalyst.

[0003] Further prior art became known from WO 2005/046857 A1 which relates to a reactor for carrying out catalyzed liquid reactions, whereby the catalyst is finely distributed in the reaction area. According to the invention, the reactor comprises at least one inlet and one outlet. All educts are supplied via the inlet and all products are discharged via the outlet. The inlet and outlet are provided with a means enabling the inlet and outlet to be connected in such a way that an outlet can be used as an inlet and at the same time an exit previously used as an outlet can be used as input used for an inlet. A filter element is provided at the inlet and outlet in order to retain the catalyst inside the reactor. Furthermore, the reactor comprises a device to ensure homogeneous distribution of the catalyst and educts in the reactor. Said reactor makes it possible to conduct reactions of the above variety with a high solid content without being obliged to separate the catalyst from the production flow in an extra procedural step and without regular cleaning and interrupting the process.

[0004] The publication CN 106220481 A discloses a technique for continuously producing polyformaldehyde dimethyl ether. The technique comprises the steps: completely dissolving methylal and trioxymethylene or polyformaldehyde to form a uniform stable raw material

mixture, pumping the raw material mixture into a solid-catalyst-filled reactor, carrying out reaction while controlling the discharge rate and feed rate to be consistent, introducing the reaction product-unreacted raw material mixture into a product collection tank, and separating the mixture from the catalyst through a stainless steel cylinder at the outlet of the reactor; and pumping the mixture in the product collection tank into a separation device, separating from the reaction product PODE2-8 the unreacted raw material to send it back into the raw material tank for cyclic utilization. The technique enables a simple preparation process, low energy consumption and continuous production.

[0005] Further WO2012011142 describes the use of CVD for manufacturing the filtration layer of membranes.

SUMMARY OF THE INVENTION

[0006] To solve technical problems occurred in continuously operating a heterogeneous reaction in a tank reactor and to fully make use of the long catalytic life of a catalyst, the present invention provides a continuous slurry-bed tank reactor as defined in claim 1 and optionally in claims 2 to 3.

[0007] In essential, the present invention provides a slurry-bed tank reactor in which a nanomaterial-based catalyst and reactants are directly separated. The present invention is applicable to catalysts with small particle sizes, even raw molecular sieve powder catalysts. Liquid materials and a catalyst are uniform slurries in the reactor, and reactants are fed into and discharged from the reactor in an uninterrupted and continuous operation manner.

[0008] The present invention is achieved by the following technical solutions.

[0009] A continuous slurry-bed tank reactor comprises a tank reactor body, an agitation system and tubular separation membranes.

[0010] The tank reactor body is respectively equipped with a thermocouple for measuring an internal temperature of the tank reactor body, a liquid level gauge for measuring a liquid level inside the tank reactor body, a third pressure gauge for measuring the pressure inside the tank reactor body, and a gas flow controller for measuring a gas flow rate into the tank reactor body and for filling nitrogen gas in the tank reactor body.

[0011] The agitation system comprises an agitation motor, an agitation shaft and an agitation impeller. The agitation motor is set up at the center over the tank reactor body. A rotor of the agitation motor is vertically downwards installed. One end of the agitation shaft is fixedly connected with the rotor of the agitation motor, and the other end thereof penetrates through the upper cap of the tank reactor body and extends to the bottom of the tank reactor body. The agitation impeller is located at the bottom of the agitation shaft.

[0012] The tubular separation membranes comprise an A tubular separation membrane and a B tubular separation membrane, which are respectively vertically downwards positioned on two sides of the agitation shaft. A first opening and a second opening of the continuous slurry-bed tank reactor are respectively communicated with two transversely symmetrical ports of a four-way valve. The A tubular separation membrane and the B tubular separation membrane are respectively communicated with two longitudinally symmetrical ports of the four-way valve. A liquid feed pump is set up at the first opening and connected with a first counterbalance valve in parallel with a first pressure gauge installed at the first counterbalance valve. A second counterbalance valve is located near the second opening, and in a pipeline communicating with the second counterbalance valve and an upper port of the four-way valve.

[0013] The four-way valve is configured to run in a time switching mode for switching the A tubular separation membrane or the B tubular separation membrane that are corresponding to the reaction mixture inlet and outlet so as to alternatively direct the reaction mixture flow into or out of the A tubular separation membrane and the B tubular separation membrane, so that the first opening and the second opening do not simultaneously feed or discharge liquid.

[0014] Both the A tubular separation membrane and the B tubular separation membrane utilize stainless steel tubes fabricated in a powder metallurgy manner as a substrate, and the permeabilities of the A tubular separation membrane and the B tubular separation membrane are finely adjusted through chemical vapor deposition thereby meeting double demands of permeability and effective separation of solid catalysts with different particle sizes and liquid reactants

[0015] In addition, at least two auxiliary material spargers are installed at a lower portion of a side wall of the tank reactor body, and each one extends into the tank reactor body, is set up at a different position in the tank reactor body, and is communicated with an auxiliary material feed pump through a pipeline.

[0016] Compared with the prior art, the present invention has the following beneficial effects:

1. 1, a slurry-bed reactor can allow a molecular sieve catalyst with small particle size fully show its excellent catalytic performance as a result of significantly weakening, and even eliminating the internal diffusion effect, thereby realizing high conversion of raw materials and high selectivity to products;
2. 2, a conventional solid-liquid separation process is omitted by utilizing tubular separation membranes that the reactants and the catalyst are directly separated in the tank reactor so as to improve the working efficiency;
3. 3, in a dual-mode time division countercurrent flow mode, adhesion of the solid catalyst to the tubular separation membranes is surmounted, the time of continuous and stable operation is prolonged, and the long catalytic life of the catalyst can be effectively reflected.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] In the drawings:

FIG. 1 is a schematic structural diagram of a continuous slurry-bed tank reactor;

FIG. 2 is a schematic diagram of a continuous slurry-bed tank reactor with a function of feeding auxiliary materials.

DESCRIPTION OF THE EMBODIMENTS

[0018] The following further describes the present invention in detail with reference to the accompanying drawings and embodiments.

[0019] As shown in FIG. 1, a continuous slurry-bed tank reactor comprises a tank reactor body 8, an agitation system 7 and tubular separation membranes.

[0020] The tank reactor body 8 is equipped with a thermocouple 10 for measuring the reaction mixture temperature inside the tank reactor body 8, a liquid level gauge 5 for measuring the liquid level inside the tank reactor body 8, a third pressure gauge 12 for measuring the internal pressure of the tank reactor body 8, and a gas flow controller 11 for measuring and controlling the gas flow rate into the tank reactor body 8.

[0021] The agitation system 7 comprises an agitation motor, an agitation shaft and an agitation impeller. The agitation motor is set up at the center over the tank reactor body 8. A rotor of the agitation motor is vertically downwards installed. One end of the agitation shaft is fixedly connected with the rotor of the agitation motor, and the other end thereof penetrates through the upper cap of the tank reactor body 8 and extends to the bottom of the tank reactor body 8. The agitation impeller is installed at the bottom of the agitation shaft.

[0022] The tubular separation membranes comprise an A tubular separation membrane 9 and a B tubular separation membrane 6, which are respectively vertically downwards located on the two sides of the agitation shaft. The B tubular separation membrane 6 and the A tubular separation membrane 9 are installed in the continuous tank reactor to achieve solid-liquid separation. The tubular separation membranes 6 and 9 utilize stainless steel tubes fabricated in a powder metallurgy manner as the substrates, and their permeability is finely adjusted through chemical vapor deposition, thereby meeting double demands of good permeability and effective separation of solid catalysts with different particle sizes and liquid reactants. The first and the second openings of the continuous slurry-bed tank reactor are respectively communicated with two transversely symmetrical ports of the four-way valve 3. The A tubular

separation membrane 9 and the B tubular separation membrane 6 are respectively communicated with two longitudinally symmetrical ports of the four-way valve 3. The first opening and the second opening do not simultaneously feed or discharge liquid. A liquid feed pump 13 is positioned at the first opening and connected with the first counterbalance valve 14 in parallel, and the first pressure gauge 15 is installed at the first counterbalance valve 14. The second counterbalance valve 2 is installed at the second opening and used for adjusting the back pressure of the continuous slurry-bed tank reactor. The pressure of the second counterbalance valve 2 is set to a value higher than the sum of vapor pressures of system mediums by 0.2-1.0 MPa. The pressure of the second counterbalance valve 2 is set to the value lower than the designed pressure of the continuous slurry-bed tank reactor to avoid overpressure of the system when the system is abnormal and/or the reactants return before the feed pump. The second pressure gauge 1 is installed in a pipeline joining the second counterbalance valve 2 and the upper port of the four-way valve 3. The second pressure gauge 1 displays the back pressure of the continuous slurry-bed tank reactor, the third pressure gauge 12 shows its internal pressure, and the first pressure gauge 15 displays its front pressure.

[0023] The four-way valve 3 is used for switching the A tubular separation membrane 9 or the B tubular separation membrane 6 that are corresponding to the reaction mixture inlet and outlet so as to alternatively direct the reaction mixture flow into or out of the A tubular separation membrane 9 and the B tubular separation membrane 6, and thereby, to clean the catalyst adhered to the tubular separation membranes by the reverse reaction mixture flow and ensure that the A tubular separation membrane 9 and the B tubular separation membrane 6 are unblocked during the whole catalytic life of a catalyst.

[0024] The four-way valve 3 generally runs in a timing switching mode. Nonetheless, when the internal pressure of the tank reactor body is increased to 0.8 MPa, the four-way valve 3 also switches the flow direction.

[0025] When the continuous slurry-bed tank reactor runs, the liquid level is maintained between a reasonable upper limit and a lower limit, and the top portion of the tank reactor body 8 is filled with nitrogen gas. In the normal running process, reactants are fed into the continuous slurry-bed tank reactor at the preset flow rate. When the liquid level is relatively high, the filled nitrogen gas flow is speeded up for increasing the internal pressure of the continuous slurry-bed tank reactor. On the contrary, if the liquid level is relatively low, the filled nitrogen gas flow rate is lowered down.

[0026] When the liquid level of the continuous slurry-bed tank reactor is beyond the preset upper limit, the four-way valve 3 switches the flow paths.

[0027] When the flow path switching time interval of the four-way valve 3 is shorter than or equal to 2 hours, the liquid feed pump 13 stops operating, and the filling of the nitrogen gas is simultaneously stopped. After the permeability of the tubular separation membranes is confirmed to be declined, the continuous slurry-bed tank reactor is turned off, and the tubular

separation membranes are replaced.

[0028] The liquid level of the reaction mixture in the tank reactor body is kept in an appropriate range by adapting the pressure of the nitrogen gas inside the tank reactor body to the feeding and discharging rates of the reaction mixture.

[0029] Further, as is shown in FIG. 2, at least two auxiliary material spargers 17 are installed at the low portion of the side wall of the tank reactor body 8, and each one extends into the tank reactor body 8, is set up at a different position in the tank reactor body 8, and communicated with one auxiliary material feed pump 16 through a pipeline.

[0030] The claimed continuous slurry-bed tank reactor is configured to run with a method comprising the following steps:

S1: adding a catalyst into the tank reactor body 8, and filling nitrogen gas in the tank reactor body 8 by the gas flow controller 11 until all air in the tank reactor body 8 is replaced with the nitrogen gas, wherein the gas flow controller 11 measures and controls the flow rate of the nitrogen gas filled in the tank reactor body 8;

S2: feeding reactants into the tank reactor body 8 sequentially through the liquid feed pump 13, the four-way valve 3 and the A tubular separation membrane 9; using a liquid level gauge to measure the liquid level of the fed reaction mixture and ensure that a distance between the liquid level of the reaction mixture and the top cap of the tank reactor body 8 is in the range of 5-80 cm; presetting the opening pressure of the first counterbalance valve 14 to a value lower than the designed pressure of the tank reactor body 8 by 0.3-0.5 MPa;

S3: when the liquid level of the reaction mixture is equal to the preset lower limit, stopping feeding the reactants; opening the heating system in the tank reactor body 8 to raise the internal temperature of the tank reactor body 8 to the designed temperature; starting the agitation system 7; determining the internal pressure of the tank reactor body 8 at this moment to be the initial pressure, and setting the opening pressure of the second counterbalance valve 2 to a value higher than the initial pressure by 0.3-0.8 MPa; maintaining the temperature for 1-3 hours after the internal temperature of the tank reactor body 8 is increased to the preset temperature; turning on the liquid feed pump 13 again to start continuously feeding the reactants;

S4: setting the four-way valve 3 to switch flow paths once every 4-8 hours for changing the direction of the reaction mixture flowing through the tubular separation membranes, wherein the four-way valve 3 switches the flow paths, and the switching time is concurrently reset when the internal pressure of the tank reactor body 8 is 0.8-1.0 MPa higher than the initially designed pressure; stopping feeding the reaction mixture when the flow path switching time interval is shorter than 2 hours due to an increase of the internal pressure of the tank reactor body 8.

[0031] Further, in step S2, at least two auxiliary material spargers 17 are installed at the lower portion of the side wall of the tank reactor body 8. The auxiliary material feed pump 16 adds reactants with poor miscibility in the tank reactor body 8 through the auxiliary material spargers 17.

[0032] Further, in step S3, when the liquid feed pump 13 starts continuously feeding the reactants, the gas flow controller 11 is turned on. First, a small flow of nitrogen gas is filled. Then, the flow rate of the nitrogen gas is dynamically adjusted in terms of the changes of the liquid level. When the liquid level of the reaction mixture is close to the liquid level upper limit, the flow rate of the nitrogen gas is enlarged. On the contrary, if the liquid level of the reactants is approaching to the liquid level lower limit, the flow rate of the nitrogen gas is decreased. Notably, when the liquid level of the reaction mixture exceeds the liquid level upper limit, the four-way valve 3 is triggered to switch the flow paths. If the flow path switching time interval of the four-way valve 3 is shorter than 2 hours due to the increase of the internal pressure of the tank reactor body, the filling of nitrogen gas by the gas flow controller 11 would be stopped.

[0033] Further, the feeding amount of the liquid feed pump 13 is determined to be 0.3-50 h^{-1} of the weight hourly space velocity on the basis of the reactant conversion and the product selectivity. When the flow path switching time interval of the four-way valve 3 is shortened due to the increase of the internal pressure or the change of the liquid level, the feeding amount of the reaction mixture is reduced until the catalytic performance of the catalyst or the permeability of the tubular separation membranes cannot meet the demands.

[0034] The following describes the current invention in detail by means of embodiments:

Embodiment 1

[0035] A quantity of 2 kg MCM-22 molecular sieve catalyst with a Si/Al molar ratio of 100 is added in a 50 L tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Liquid reactants of dimethoxymethane and 1 ,3,5-trioxane with a molar ratio of 2:1 are pumped into the tank reactor by a liquid feed pump 13 up to the distance between the liquid level and the top cap of the tank reactor body 8 of 15 cm. Then, the heating system of the tank reactor is started to raise the temperature inside the tank reactor body 8 to 50 °C. Simultaneously, the agitation system 7 is initiated. After two hours, the liquid feed pump 13 restarts to continuously feed the reaction mixture. The opening pressure of counterbalance valve 2 is set to 0.5 MPa; the flow rate of the liquid feed pump 13 is set to 30 l/h; the flow path switching time interval of the four-way valve 3 is set to six hours; the starting pressure of counterbalance valve 14 is set to 1.2 MPa, and the initial flow rate of the gas flow controller 11 is set to 5 sccm (1/12 ml/s) that is later altered according to the changes of the liquid level in the following way. The flow rate of the gas flow controller 11 is increased with the elevation of the liquid level, but is decreased with the reduction of the liquid level. In the present embodiment, to maintain normal operation, the liquid level needs to intermediate between the upper limit of 5 cm and the lower limit of 15

cm distant from the top cap of the tank reactor body 8.

[0036] The tank reactor continuously and stably runs. In the later process, the conversion of the reactants lowers down due to the deactivation of the catalyst. Thereafter, the reaction is stopped.

Embodiment 2

[0037] A quantity of 2 kg MCM-22 molecular sieve catalyst with a Si/Al molar ratio of 200 is added in a 50 l tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Dimethoxymethane and 1,3,5-trioxane liquid mixture with a molar ratio of 2.5:1 are pumped into the tank reactor by the liquid feed pump 13 up to the distance between the liquid level and the top cap of the tank reactor body 8 of 15 cm. After that, the heating system of the tank reactor is opened to elevate the temperature of the tank reactor to 65 °C. Simultaneously, the agitation system 7 is started. After one hour, the liquid feed pump 13 restarts to continuously feed the reactants. The opening pressure of counterbalance valve 2 is set to 0.8 MPa; the flow rate of the liquid feed pump 13 is set to 50 l/h; the flow path switching time interval of the four-way valve 3 is set to 4 hours; the opening pressure of counterbalance valve 14 is set to 1.2 MPa, and the initial flow rate of the gas flow controller 11 is set to 5 sccm that is adjusted in terms of the liquid level in the reaction process in the following way. The flow rate of the gas flow controller 11 is increased with the elevation of the liquid level, and is decreased with the decline of the liquid level. In the present embodiment, to maintain normal operation, the upper limit and the lower limit of the liquid level are set to 5 and 15 cm from the top cap of the tank reactor body 8 respectively.

[0038] The tank reactor continuously and stably runs for more than 500 hours. In the later process, the conversion of the raw materials lowers down, but the selectivity to product is still high.

Embodiment 3

[0039] A quantity of 2 kg MCM-22 molecular sieve catalyst with a Si/Al molar ratio of 50 is added in a 50 l tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Dimethoxymethane and 1,3,5-trioxane liquid mixture with a molar ratio of 2.5:1 are pumped into the tank reactor by a liquid feed pump 13 until the distance between the liquid level and the top cap of the tank reactor body 8 reaches 15 cm. Then, the heating system of the tank reactor is started to raise the temperature of the tank reactor to 35 °C. Simultaneously, the agitation system 7 is initiated. After 3 hours, the liquid feed pump 13 restarts to continuously feed the reactants. The opening pressure of the counterbalance valve 2 is set to 0.3 MPa; the flow rate of the liquid feed pump 13 is set to 50 l/h; the flow path switching time interval of the four-way valve 3 is set to eight hours; the opening pressure of the counterbalance valve 14 is

set to 1.2 MPa, the initial flow rate of the gas flow controller 11 is set to 5 sccm (1/12 ml/s) that is adjusted according to the changes of the liquid level in the reaction process in the following way. The flow rate of the gas flow controller 11 is increased with the elevation of the liquid level, and is decreased with the decline of the liquid level. In the present embodiment, to maintain normal operation, the upper limit and the lower limit of the liquid level are set to 5 and 15 cm from the top cap of the tank reactor body 8 respectively.

[0040] After the tank reactor continuously runs for more than 100 hours, the conversion of the reactants is slightly decreased although the selectivity to products is still high.

Embodiment 4

[0041] A quantity of 2 kg MCM-22 molecular sieve catalyst with a Si/Al molar ratio of 50 is added in a 50 l tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Liquid reactants of dimethoxymethane and 1 ,3,5-trioxane with a molar ratio of 2.5:1 are pumped into the tank reactor by a liquid feed pump 13 until the distance between the liquid level and the top cap of the tank reactor body 8 reaches 15 cm. Afterwards, the heating system of the tank reactor is started to raise the temperature of the tank reactor to 35 °C. Simultaneously, the agitation system 7 is initiated. After 3 hours, the liquid feed pump 13 restarts to continuously feed the reactants. The opening pressure of counterbalance valve 2 is set to 0.3 MPa; the flow rate of the liquid feed pump 13 is set to 50 l/h; the flow path switching time interval of the four-way valve 3 is set to eight hours; the opening pressure of counterbalance valve 14 is set to 1.2 MPa, and the initial flow rate of a gas flow controller 11 is set to 5 sccm (1/12 ml/s) that is adjusted according to the changes of the liquid level in the reaction process in the following way. The flow rate of the gas flow controller 11 is enlarged with the elevation of the liquid level, and is decreased with the reduction of the liquid level. In the present embodiment, to maintain normal operation, the upper limit and the lower limit of the liquid level are set to 5 and 15 cm from the top cap of the tank reactor body 8.

[0042] The tank reactor continuously and stably runs. In the later process, the conversion of the reactants is slightly decreased due to the deactivation of the catalyst, thus the reaction is stopped.

Embodiment 5

[0043] A quantity of 3 kg titanium silicalite molecular sieve catalyst is added to a 50 l tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Benzene is pumped into the tank reactor by the liquid feed pump 13 until the distance between the liquid level and the top cap of the tank reactor body 8 reaches 15 cm. Afterwards, the heating system of the tank reactor is opened to raise the temperature of the tank reactor to 85 °C. Simultaneously, the agitation system 7 is started. After three hours, the liquid feed pump 13 restarts to continuously

feed the reactants. Hydrogen peroxide is pumped through the auxiliary material feed pump 16, and fed into the tank reactor by four auxiliary material spargers 17 at a rate of 5 l/h in different positions. The auxiliary material feed pump 16 and the liquid feed pump 13 are controlled to simultaneously start and stop. The opening pressure of the counterbalance valve 2 is set to 0.6 MPa; the flow rate of the liquid feed pump 13 is set to 20 l/h; the flow path switching time interval of the four-way valve 3 is set to four hours; the opening pressure of the counterbalance valve 14 is set to 1.8 MPa, the initial flow rate of the gas flow controller 11 is set to 5 sccm (1/12 ml/s) that is adjusted in terms of the changes of the liquid level in the reaction process in the following way. The flow rate of the gas flow controller 11 is increased with the elevation of the liquid level, and is decreased with the decline of the liquid level. In the present embodiment, to maintain normal operation, the upper and the lower limits of the liquid level are set to 10 and 20 cm from the top cap of the tank reactor body 8 respectively. When the distance between the liquid level and the top cap of the tank reactor body 8 is shorter than 10 cm, the auxiliary material feed pump 16 and the auxiliary material spargers 17 are stopped.

Embodiment 6

[0044] A quantity of 3 kg titanium silicalite molecular sieve catalyst is added in a 50 l tank reactor. Nitrogen gas is filled to replace air in the tank reactor. Cyclohexanone is pumped into the tank reactor by a liquid feed pump 13 up to the distance between the liquid level and the top cap of the tank reactor body 8 of 15 cm. Afterwards, the heating system of the tank reactor is started to raise the temperature of the tank reactor to 75 °C. Simultaneously, the agitation system 7 is initiated. After two hours, the liquid feed pump 13 restarts to continuously feed the reactants. Hydrogen peroxide is pumped through the auxiliary material feed pump 16, and fed into the tank reactor by four auxiliary material spargers 17 at a rate of 10 l/h in different positions. The auxiliary material feed pump 16 and the liquid feed pump 13 are controlled to simultaneously start and stop. The opening pressure of the counterbalance valve 2 is set to 0.6 MPa; the flow rate of the liquid feed pump 13 is set to 30 l/h; the flow path switching time interval of the four-way valve 3 is set to four hours; the opening pressure of the counterbalance valve 14 is set to 1.6 MPa, and the initial flow rate of the gas flow controller 11 is set to 5 sccm (1/12 ml/s) that is adjusted according to the changes of the liquid level in the following way. The flow rate of the gas flow controller 11 is increased with the elevation of the liquid level, and is decreased with the reduction of the liquid level. In the present embodiment, to maintain normal operation, the upper and the lower limits of the liquid level is 8 and 20 cm away from the top of the tank reactor body 8, respectively. When the distance between the liquid level and the top cap of the tank reactor body 8 is less than 8 cm, the auxiliary material feed pump 16 and the auxiliary material spargers 17 are stopped.

[0045] The above merely describes specific embodiments of the present invention, but the protection scope of the present invention is not limited thereto. A person skilled in the art can easily conceive modifications or replacements within the technical scope of the present invention, which is defined by the claims.

Listing of reference numerals

[0046]

1 second pressure gauge,
2 second counterbalance valve,
3 four-way valve,
4 liquid level,
5 liquid level gauge,
6 B tubular separation membrane,
7 agitation system,
8 tank reactor body,
9 A tubular separation membrane,
10 thermocouple,
11 gas flow controller,
12 third pressure gauge,
13 liquid feed pump,
14 first counterbalance valve,
15 first pressure gauge,
16 auxiliary material feed pump,
17 auxiliary material sparger.

REFERENCES CITED IN THE DESCRIPTION

Cited references

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Patent documents cited in the description

- CN2016105989527 **[0002]**
- CN106220481A **[0002]** **[0004]**
- WO2005046857A1 **[0003]**
- WO2012011142A **[0005]**

Patentkrav

1. Kontinuerlig slambeholderreaktor, omfattende et tankreaktorlegeme (8), et omrøringssystem (7) og rørformede separationsmembraner, hvor

tankreaktorlegemet (8) er udstyret med henholdsvis et termoelement (10) til måling af den indvendige temperatur i tankreaktorlegemet (8), en væskeniveaumåler (5) til måling af et væskeniveau inde i tankreaktorlegemet (8), en tredje trykmåler (12) til måling af et indvendigt tryk i tankreaktorlegemet (8) og en gasstrømningsregulator (11) til måling og styring af en gasstrømningshastighed ind i tankreaktorlegemet (8);

omrøringssystemet (7) omfatter en omrøringsmotor, en omrøringsaksel og et omrøringsskovlhjul;

omrøringsmotoren er opstillet i midten over tankreaktorlegemet (8); en rotor af omrøringsmotoren er installeret lodret nedad;

den ene ende af omrøringsakslen er fast forbundet med rotoren på omrøringsmotoren, og den anden ende deraf trænger gennem det øvre dæksel af tankreaktorlegemet (8) og strækker sig til bunden af tankreaktorlegemet (8); omrøringshjulet er placeret i bunden af omrøringsakslen;

de rørformede adskillelsesmembraner omfatter en rørformet adskillelsesmembran A (9) og en rørformet adskillelsesmembran B (6), som hver især er anbragt lodret nedad på to sider af omrøringsakslen;

en første åbning og en anden åbning af den kontinuerlige slambeholderreaktor er hver især forbundet med to tværsymmetriske porte på en firevejsventil (3);

den rørformede separationsmembran A (9) og den rørformede adskillelsesmembran B (6) er hver især forbundet med to langsgående symmetriske porte på firevejsventilen (3); den første åbning og den anden åbning tilfører eller udleder ikke væske samtidigt; **kendetegnet ved, at**

en væsketilførselspumpe (13) er opstillet ved den første åbning og forbundet med en første modvægtsventil (14) parallelt med en første trykmåler (15), der er installeret ved den første modvægtsventil (14);

en anden modvægtsventil (2) er installeret ved den anden åbning, og en anden trykmåler (1) er installeret i en rørledning, der står i forbindelse med den anden modvægtsventil (2) og en øvre port på firevejsventilen (3);

firevejsventilen (3) er konfigureret til at skifte strømningsveje for at ændre retninger af væske, der strømmer gennem den rørformede adskillelsesmembran A (9) eller den rørformede adskillelsesmembran B (6) for alternativt at lede en reaktionsblandingsstrøm ind eller ud af den rørformede adskillelsesmembran A (9) og den rørformede adskillelsesmembran B (6) ved generelt at køre firevejsventilen (3) i en tidsskiftningstilstand for at rense katalysatoren, der klæber til den rørformede adskillelsesmembran A eller den rørformede adskillelsesmembran B (9, 6) ved omvendt reaktionsblandingsstrøm og også skift af strømningsretning, når det indre tryk af tankreaktorlegemet (8) øges til 0,8 MPa;

slambeholderreaktoren er således konfigureret, at en nanomateriale-baseret katalysator og reaktanter kan adskilles direkte, hvor både den rørformede adskillelsesmembran A (9) og den rørformede adskillelsesmembran B (6) anvender rustfrie stålrør, der er fremstillet på en pulvermetallurgisk måde som et substrat, og permeabiliteterne af den rørformede adskillelsesmembran A (9) eller den rørformede adskillelsesmembran B (6) finjusteres gennem kemisk dampaflejring, hvorved dobbelte krav til permeabilitet og effektiv adskillelse af faste katalysatorer med forskellige partikelstørrelser og flydende reaktanter opfyldes, hvor

slambeholderreaktoren er konfigureret til at køre følgende trin:

S1: tilføjelse af en katalysator til tankreaktorlegemet og påfyldning af nitrogengas i tankreaktorlegemet ved hjælp af gasstrømningsregulatoren, indtil al luft i tankreaktorlegemet er erstattet med nitrogengas, hvor gasstrømsregulatoren måler og styrer strømningshastigheden af nitrogengas, der fyldes i tankreaktorlegemet;

S2: tilførsel af reaktanter ind i tankreaktorlegemet sekventielt gennem væsketilførselspumpen, firevejsventilen og den rørformede separationsmembran A; anvendelse af en væskeniveaumåler til at måle væskeniveauet af de tilførte reaktanter og sikre, at en afstand mellem reaktanternes væskeniveau og det øvre dæksel på tankreaktorlegemet er i området 5-80 cm; forudindstilling af åbningstrykket for den første modvægtsventil til en værdi lavere end det beregnede tryk for tankreaktorlegemet med 0,3-0,5 MPa;

S3: når væskeniveauet i reaktanterne når den forudindstillede nedre grænse, standsning af tilførsel af reaktanterne; åbning af et varmesystem i tankreaktorlegemet for at hæve den indvendige temperatur af tankreaktorlegemet til den forudindstillede temperatur; samtidig åbning af omrøringssystemet; bestemmelse af det indvendige tryk af tankreaktorlegemet på dette tidspunkt som starttrykket og indstilling af åbningstrykket af den anden modvægtsventil til at være 0,3-0,8 MPa højere end starttrykket; opretholdelse af temperaturen i 1-3 timer efter, at den indvendige temperatur af tankreaktorlegemet er hævet til den forudindstillede temperatur; tænding af væsketilførselspumpen igen for at begynde kontinuerligt at tilføre reaktanterne;

S4: indstilling af firevejsventilen til at skifte strømningsveje en gang hver 4.-8. time for at ændre retningerne af reaktionsblandingen, der strømmer gennem de rørformede adskillelsesmembraner; nulstilling af firevejsventilen for at skifte strømningsvejene og genberegning af strømningsvejsskiftetiden, når det indvendige tryk i tankreaktorlegemet er højere end starttrykket med 0,8-1,0 MPa; standsning af tilførsel af reaktanterne, når tidsintervallet for at skifte strømningsvejen er kortere end to timer på grund af stigningen i det indre tryk i tankreaktorlegemet.

2. Kontinuerlig slambeholderreaktor ifølge krav 1, hvor mindst to hjælpematerialespredere (17) er installeret ved den nedre del af en sidevæg af tankreaktorlegemet (8); og hver enkelt strækker sig ind i tankreaktorlegemet (8) og er forbundet med en hjælpematerialefødepumpe (16) gennem en rørledning.

3. Kontinuerlig slambeholderreaktor ifølge krav 1, hvor firevejsventilen (3), der kører i tidsomskiftningstilstanden, har en yderligere strømningsskifttilstand på grund af væskeniveau, når væskeniveauet i reaktionsblandingen overstiger en øvre væskeniveaugrænse for tankreaktorlegemet (8).

DRAWINGS

Drawing

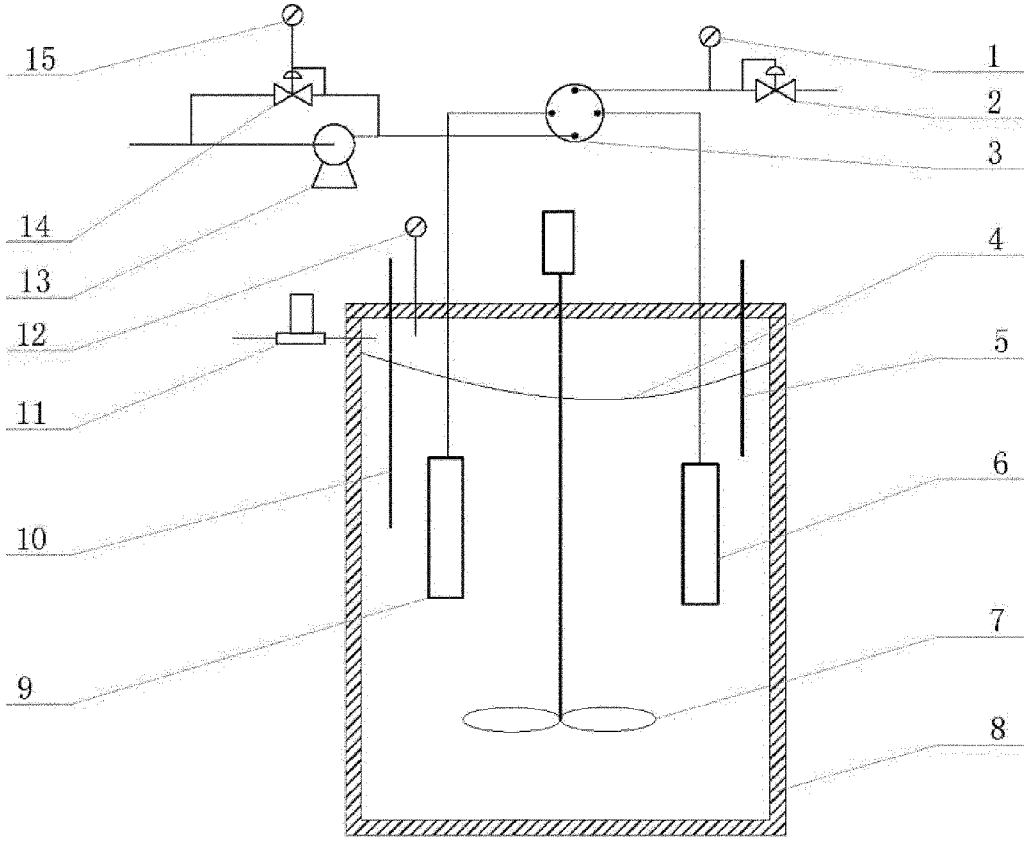

Fig. 1

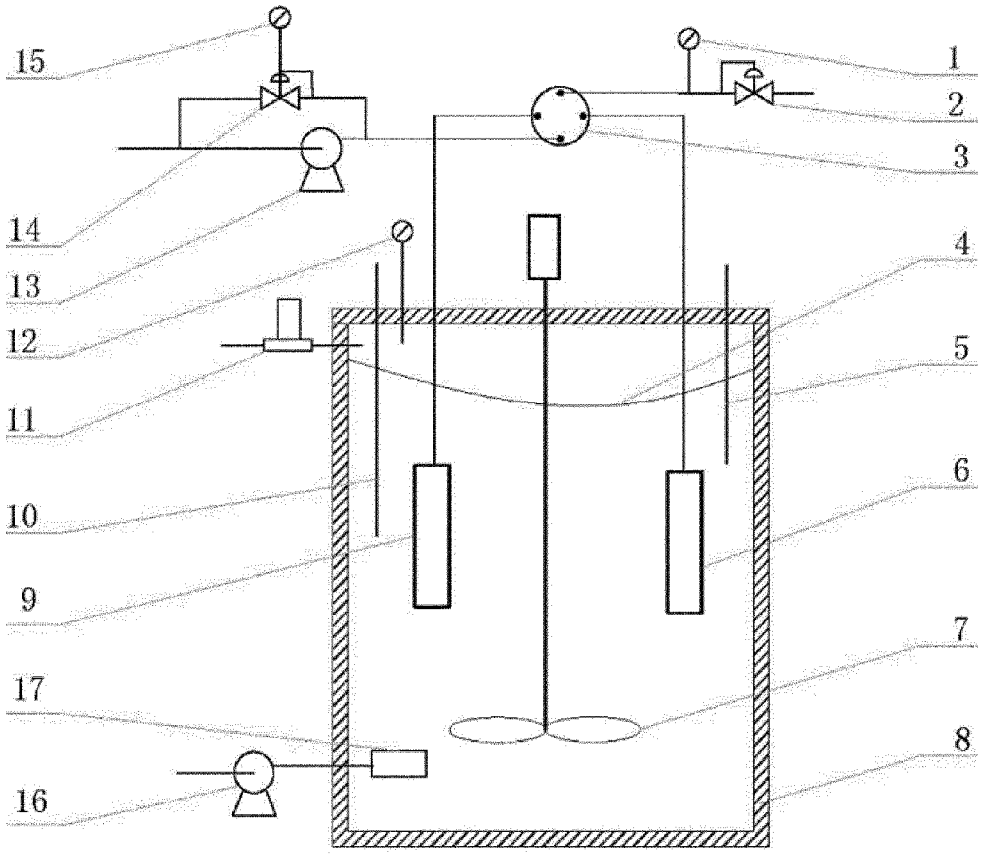

Fig. 2