

METHOD AND SYSTEM FOR SEPARATING T-BUTANOL FROM DIISOBUTYLENE

TECHNICAL FIELD

The present invention belongs to the field of chemical industry and organic synthesis and relates to the separation of tert-butanol from diisobutylene.

BACKGROUND ART

Diisobutylene (DIB, 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene) can be prepared by isobutylene oligomerization and catalysts are generally acidic ion exchange resins and molecular sieves, etc. DIB is a versatile chemical intermediate that can be hydrogenated to produce isooctane which is an additive that increases the octane number of gasoline; DIB can react with phenol to synthesize octylphenol which is further used in the production of antioxidants, tackifying resins and surfactants and other products; DIB also can be hydroformylated to produce isononylaldehyde. Isononylaldehyde is hydrogenated to produce isononanol and oxidized to produce isononanoic acid, and further used in the production of plasticizers, lubricants and other products.

In the process of acid catalyst catalyzing isobutylene oligomerization to produce DIB, the addition of tert-butanol (TBA) can significantly inhibit the trimerization and high polymerization of isobutylene, thereby increasing the selectivity of DIB. TBA is not consumed during the reaction and needs to be recycled after being separated from the product. However, TBA is azeotropic with DIB, and conventional distillation methods cannot separate them. The aqueous extraction of the TBA/DIB mixture is the most easily achieved separation method. The oil phase gives DIB and the aqueous phase gives a substantially DIB-free aqueous solution of TBA. However, TBA is also azeotropic with water and it is difficult to recycle TBA contained in the extracted aqueous phase to obtain pure TBA. The preparation of anhydrous TBA with aqueous TBA generally employs the methods such as recrystallization or salting out, etc, wherein recrystallization is a well-known high energy consumption operation in the art. There are also literatures (potassium carbonate salting out of tert-butanol-water system and industrialization test, Han Pingfang et al., Chemical Progress, Vol.23, No.8, 2004, P877-879) employing salting out method to separate the azeotrope of TBA and water. However, the TBA obtained by

salting out will inevitably carry metal ions. If it is recycled directly to the isobutylene oligomerization reactor, it will poison the catalyst. US Patent No. 6,734,333 uses liquefied isobutene to extract TBA from water; due to the good compatibility of TBA and water, the amount of isobutylene in liquefied isobutylene used to extract TBA must be very large, which inevitably results in a large size of the extraction tower; and if the isobutylene is to be liquefied, the extraction tower must be pressurized (at least 0.3 MPa gauge pressure at room temperature to liquefy isobutylene). Extraction towers, especially rotary extraction towers containing moving equipment, have both large investment and safety risks. In order to avoid the above problem, US Patent No. 6,863,778 employs two towers for pressure swing distillation to separate DIB from TBA. The pressure of the high pressure tower is 0.5-0.9 MPa and the pressure of the low pressure tower is 0-0.3 MPa; but the pressure distillation makes the tower more energy-consuming and at the same time, the investment in equipment is also greatly increased.

Therefore, there is a need to find a method that does not introduce a liquid extraction agent, has mild operating conditions and low energy consumption and can achieve separation of TBA from DIB to solve various problems existing in the prior art.

SUMMARY OF THE INVENTION

The object of the present invention is to separate tert-butanol from diisobutylene under the condition that no extraction agent is introduced and the energy consumption for separation is effectively reduced.

To achieve the above object, the technical solution adopted by the present invention is as follows:

In one aspect, the present invention provides a method for separating tert-butanol from diisobutylene, comprising:

(1) passing TBA-DIB azeotrope and an adsorbing agent through a second ejector into a suspended bed to perform an adsorption process, and discharging the adsorption product from the outlet of the suspended bed into a first separator for separation, wherein the obtained material liquid rich in DIB is discharged from the upper outlet of the first

separator and the adsorbing agent which has adsorbed TBA is discharged from the lower outlet of the first separator;

(2) introducing the adsorbing agent which has adsorbed TBA and a desorption gas into a second separator through a first ejector for separation, wherein the obtained gas rich in TBA vapor is discharged from the upper outlet of the second separator, and the adsorbing agent which has desorbed TBA is discharged from the lower outlet of the second separator.

To achieve the above method, in another aspect, the present invention provides a system for separating tert-butanol from diisobutylene, comprising:

a suspended bed, wherein the feed pipe of the suspended bed is connected to a DIB-TBA azeotrope supply pipe through a second ejector, and the outlet of the suspended bed is connected to the inlet of a first separator;

a first separator, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the suspended bed, the upper outlet is used for discharging material liquid rich in DIB and the lower outlet is connected to a second separator through a first ejector;

a second separator, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the first separator through the first ejector, the upper outlet is used for discharging gas rich in TBA vapor and the lower outlet is used for discharging adsorbing agent which has desorbed TBA.

According to the method of the present invention, the purity of the DIB obtained directly from the upper outlet of the first separator can reach more than 95 wt%; the purity of the TBA obtained directly by condensing the gas rich in TBA vapor at the upper outlet of the second separator can reach more than 70 wt%.

The positive effect of the present invention lies in that the TBA-DIB azeotrope is directly separated without introducing extraction agents, to obtain DIB and TBA respectively. This method can continuously perform the adsorption and desorption operations. The entire process has no large-scale dynamic equipment or high-pressure equipment and the main process has no high-temperature operating unit. This method has low investment, low energy consumption and low safety risk.

DESCRIPTION OF THE DRAWINGS

Figure 1: A schematic diagram of a separation system and its technological process with a second ejector mounted horizontally; wherein, 1 is a suspended bed, 2 is a first separator, 3 is a storage tank of the first separator, 3-1 is the upper storage tank of the first separator, 3-2 is the lower storage tank of the first separator, 4 is a first ejector, 5 is a second separator, 6 is the storage tank of the second separator, 6-1 is the upper storage tank of the second separator, 6-2 is the lower storage tank of the second separator, 7 is a second ejector, 8 is a first distillation tower, 9 is a condenser and 10 is a second distillation tower;

Figure 2: A schematic diagram of a separation system and its technological process with a second ejector mounted vertically; the meanings of the reference numbers are the same as those in Figure 1;

Figure 3: A schematic diagram of an ejector; wherein 31 is a driving gas/liquid inlet, 32 is a reducing pipe, 33 is a throat pipe, 34 is a negative pressure suction chamber sleeve, 35 is a negative pressure inlet, 36 is a mixing pipe, 37 is a diffusion pipe and 38 is the outlet of the ejector;

D1 is the diameter of the negative pressure suction chamber sleeve, D2 is the diameter of the driving gas/liquid inlet, D3 is the diameter of the throat pipe, D4 is the diameter of the mixing pipe, L1 is the length of the throat pipe, L2 is the distance between the outlet of the throat pipe and the inlet of the mixing pipe, L3 is the length of the mixing pipe, L4 is the length of the diffusion pipe, A1 is the 1/2 taper angle of the reducing pipe, A2 is the 1/2 taper angle of the junction between the mixing pipe and the negative pressure inlet, and A3 is the 1/2 taper angle of the diffusion pipe.

DETAILED DESCRIPTION OF THE INVENTION

In the method of the present invention, TBA is selectively adsorbed by an adsorbing agent flowing together with TBA and DIB in a suspended bed, and the continuous separation of DIB, TBA, and the adsorbing agent and the continuous regeneration of the adsorbing

agent are accomplished via a gas-liquid-solid centrifugal separator, a gas-solid separator and ejector nozzles driven by gas and liquid respectively, etc., thereby achieving the purpose of separating DIB from TBA. The present invention will be described in detail below with reference to specific embodiments.

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As described above, the method for separating tert-butanol from diisobutylene of the present invention comprises:

(1) passing a TBA-DIB azeotrope and an adsorbing agent through a second ejector into a suspended bed to perform an adsorption process, and discharging the adsorption product from the outlet of the suspended bed into a first separator for separation, wherein the obtained material liquid rich in DIB is discharged from the upper outlet of the first separator and the adsorbing agent which has adsorbed TBA is discharged from the lower outlet of the first separator; said material liquid rich in DIB contains a very small amount of TBA;

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(2) introducing the adsorbing agent which has adsorbed TBA and a desorption gas into a second separator through a first ejector for separation, wherein the obtained gas rich in TBA vapor is discharged from the upper outlet of the second separator, and the adsorbing agent which has desorbed TBA is discharged from the lower outlet of the second separator.

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Further, the method further comprises: (3) recycling the adsorbing agent which has desorbed TBA back to the suspended bed through the second ejector.

Preferably, in step (1), a small amount of TBA-DIB azeotrope is separated out from the material liquid rich in DIB by distillation to obtain high-purity DIB.

Preferably, in step (1), the composition of the azeotrope at atmospheric pressure is TBA:DIB=62:38 (mass ratio).

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Preferably, in step (2), gas rich in TBA vapor is condensed to obtain TBA directly; more preferably, the TBA-DIB azeotrope is further separated out by distillation from the TBA obtained by condensation to obtain high-purity TBA.

More preferably, in steps (1) and (2), the TBA-DIB azeotrope separated out by distillation is recycled back to the suspended bed.

- 5 In a preferred embodiment, the method for separating tert-butanol from diisobutylene of the present invention comprises:
- (1) passing a TBA-DIB azeotrope and an adsorbing agent through a second ejector into a suspended bed to perform an adsorption process, and discharging the adsorption product from the outlet of the suspended bed into a first separator for separation, wherein the
10 obtained material liquid rich in DIB is discharged from the upper outlet of the first separator and the adsorbing agent which has adsorbed TBA is discharged from the lower outlet of the first separator;
 - (2) introducing the adsorbing agent which has adsorbed TBA and a desorption gas into a second separator through a first ejector for separation, wherein the obtained gas rich in
15 TBA vapor is discharged from the upper outlet of the second separator, and the adsorbing agent which has desorbed TBA is discharged from the lower outlet of the second separator;
 - (3) recycling the adsorbing agent obtained in step (2) which has desorbed TBA back to the suspended bed through the second ejector;
 - 20 (4) separating out a small amount of TBA-DIB azeotrope from the material liquid rich in DIB obtained in step (1) to obtain high-purity DIB by distillation, and recycling the separated TBA-DIB azeotrope back to the suspended bed;
 - (5) separating out TBA-DIB azeotrope from the gas rich in TBA vapor obtained in step (2) to obtain high purity TBA by condensation and distillation; and recycling the separated
25 TBA-DIB azeotrope to the suspended bed.

Preferably, the adsorbing agent used in the present method is a polystyrene resin loaded with strong polar groups. The polystyrene resin is a copolymer of styrene and divinylbenzene and/or a copolymer of a copolymer of styrene and divinylbenzene
30 compounded with short fibers, preferably a copolymer of a copolymer of styrene and divinylbenzene compounded with short fibers to increase the mechanical strength of particles. The short fibers include, but are not limited to, one or more of metal fiber particles, glass fiber particles and cellulose particles.

35 Further preferably, the strong polar group is one or more of a sulfonic acid group, a nitro

group, an amino group and a carboxyl group, preferably a sulfonic acid group. The loading amount of the strong polar group is 0.5 to 10 mmol/g, preferably 3 to 5.5 mmol/g; the loading amount of the strong polar group means the mole of the strong polar group loaded in per unit mass of the adsorbing agent.

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Further preferably, the adsorbing agent consists of porous spherical particles, and has a roundness of 0 to 0.1, an average particle size of 0.1 to 5 mm, preferably 0.8 to 1.5 mm, and a uniform particle size, based on the total weight of the adsorbing agent, the adsorbing agent particles with a particle size less than 0.7 times the average particle size are not more than 5%, the adsorbing agent particles with a particle size greater than 1.3 times the average particle size are not more than 5%; and the pore size of the adsorbing agent is from 10 to 500 nm, preferably from 100 to 300 nm and the specific surface area of the adsorbing agent is from 5 to 100 m²/g, preferably 20 to 80 m²/g; the true density of the adsorbing agent is 0.8 to 1.5 g/ml, preferably 1 to 1.2 g/ml. The adsorbing agent particles with high roundness and uniform particle size are beneficial to improve the fluidity of the particles and reduce the frictional breakage between the adsorbing agent particles.

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More preferably, the average length of the short fiber is 1/100 to 1/2, preferably 1/20 to 1/5 of the average particle size of the adsorbing agent; the average diameter of the short fiber is 1/10000 to 1/5, preferably 1/1000 to 1/10 of the average length.

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Before the strong polar groups are loaded, the exposed fibers on the surface of the polystyrene resin, for example, a copolymer of a copolymer of styrene and divinylbenzene compounded with short fibers, need to be dissolved to obtain particles with smooth surfaces. Then the obtained surface-treated adsorbing agent particles are loaded with strong polar groups to obtain the polystyrene resin loaded with strong polar groups; wherein the metal fibers are dissolved by using one or more of dilute nitric acid, dilute sulfuric acid and dilute hydrochloric acid, and the glass fibers are dissolved by using hydrofluoric acid and the cellulose is dissolved by using copper amine and/or cupri-ethylenediamine.

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Preferably, in step (1), the mass ratio of tert-butanol in the TBA-DIB azeotrope to the adsorbing agent at the inlet of the suspended bed is 1:0.5 to 1:20, preferably 1:2 to 1:10.

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Further preferably, the adsorbing agent is added at one time. After reaching the specified mass ratio of tert-butanol to the adsorbing agent, the adsorbing agent is no longer added and is circulated in the system.

- 5 Preferably the TBA-DIB azeotrope and solid adsorbing agent are fed from the inlet at the bottom of the suspended bed. More preferably, the feed linear velocity of the TBA-DIB azeotrope is 1.05 to 20 times, preferably 2 to 10 times the critical suspension velocity of the adsorbing agent; and the average linear velocity of the adsorbing agent in the suspended bed is 1 to 99%, preferably 50 to 90% of the linear velocity of the TBA-DIB
10 azeotrope.

The inlet speed of the separators needs to be controlled in a suitable range. If the speed is too low, the centrifugal effect is not good. If the speed is too high, the risk of collision and fracture of the adsorbing agent is increased. Preferably, in step (1), the average linear
15 velocity of the adsorbing agent which has adsorbed TBA together with the material liquid rich in DIB entering the first separator from the outlet of the suspended bed is 1 to 50 m/s, preferably 3 to 30 m/s.

Preferably, in step (2), the gas velocity at the inlet of the second separator is 0.5-50 m/s,
20 preferably 2-10 m/s; the operating temperature is 25-60°C, preferably 30-50°C.

Preferably, the first ejector is driven by a desorption gas. The desorption gas is a non-flammable, non-combustion-supporting gas, such as one or more of nitrogen, carbon dioxide, argon, and helium, and preferably nitrogen and/or carbon dioxide. The
25 temperature of the desorption gas of the first ejector is 25-60°C, preferably 30-50°C; if the temperature is too high, it will cause tert-butanol to react on the surface of the adsorbing agent.

The second ejector is driven by a liquid and the driving liquid is TBA-DIB azeotrope; the
30 driving liquid has a maximum velocity at the throat pipe of the second ejector of 0.5 to 50 m/s, preferably 2 to 10m/s.

As shown in FIGS. 1 and 2, the present invention further provides a system for separating tert-butanol from diisobutylene, comprising:

- 35 a suspended bed 1, wherein the feed pipe of the suspended bed is connected to a

DIB-TBA azeotrope supply pipe through a second ejector 7 and the outlet of the suspended bed is connected to the inlet of a first separator 2;

5 a first separator 2, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the suspended bed 1, the upper outlet is used for discharging material liquid rich in DIB, and the lower outlet is connected to a second separator 5 through a first ejector 4;

10 a second separator 5, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the first separator 2 through the first ejector 4, the upper outlet is used for discharging gas rich in TBA vapor and the lower outlet is used for discharging adsorbing agent which has desorbed the TBA.

Preferably, the lower outlet of the second separator 5 is connected to the feed pipe of the suspended bed 1 through the second ejector 7.

15 Preferably, the upper outlet of the first separator 2 is connected to a first distillation tower 8 through a pipe. The upper outlet of the first distillation tower 8 is used for discharging TBA-DIB azeotrope and the lower outlet of the first distillation tower 8 is used for discharging high-purity DIB. Further preferably, the upper outlet of the first distillation tower 8 is connected to the TBA-DIB azeotrope supply pipe.

20 Preferably, the upper outlet of the second separator 5 is connected to a condenser 9 through a pipe, or the upper outlet of the second separator 5 is connected to the condenser 9 through a pipe and the material outlet of the condenser is connected to the inlet of a second distillation tower 10. The upper outlet of the second distillation tower is used for discharging TBA-DIB azeotrope, and the lower outlet the second distillation tower is used for discharging high-purity TBA. Further preferably, the upper outlet of the second distillation tower is connected to the TBA-DIB azeotrope supply pipe.

30 Preferably, the lower part of the first separator is a first separator storage tank 3 (a first storage tank for short), which is divided into an upper storage tank 3-1 and a lower storage tank 3-2; the lower part of the second separator is a second separator storage tank 6 (a second storage tank for short), which is divided into an upper storage tank 6-1 and a lower storage tank 6-2.

35 Preferably, in the method and system of the present invention, the suspended bed may be

any conventional suspended bed. The height-to-diameter ratio of the suspended bed is 1000:1 to 1:1, preferably 100:1 to 10:1; The operating pressure is atmospheric pressure; the operating temperature is between 25°C and the boiling point of TBA-DIB azeotrope (25-77°C).

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The inlet at the bottom of the suspended bed is an equal-diameter straight pipe or a reducing pipe or a combination thereof, preferably a reducing pipe whose taper angle gradually becomes smaller, and the taper angle is between 0 and 60°, preferably between 0 and 30°; the minimum cross-sectional area of the passage of the reducing pipe is 20 to 80%, preferably 30 to 50% of the maximum cross-sectional area. Using a reducing pipe at the inlet at the bottom of the suspended bed can form a jet at the bottom of the suspended bed, increasing the turbulence at the bottom of the suspended bed, avoiding sedimentation of the adsorbing agent at the bottom and meanwhile promoting the liquid-solid mass transfer.

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The outlet of the suspended bed is a round angle outlet or a taper angle outlet, so as to prevent the adsorbing agent from being broken when the adsorbing agent flows therethrough and directly collided with a right angle due to that the passage is suddenly reduced and the flow rate becomes larger. Preferably, the outlet of the suspended bed and the inlet of the first separator is connected by an equal-diameter straight pipe or a reducing pipe or a combination thereof, preferably a reducing pipe, and more preferably a reducing pipe whose taper angle gradually decreases, wherein the maximum taper angle does not exceed 60°, the minimum taper angle is not less than 5°, preferably between 10 and 30°; the minimum cross-sectional area of the reducing pipe is 20 to 80%, preferably 30 to 50% of the maximum cross-sectional area of the reducing pipe. Using a reducing pipe between the outlet of the suspended bed and the inlet of the first separator can increase the initial velocity of the material at the inlet of the first separator and improve the effect of centrifugal separation.

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In the method and system of the present invention, the first separator and the second separator are preferably centrifugal separators.

The first separator consists of an upper straight cylinder section, a middle conical section and a lower first storage tank. The height-to-diameter ratio of the straight cylinder section is 1:1 to 10:1, preferably 2:1 to 4:1; the taper angle of the conical section is 3 to 30°,

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preferably 5 to 15°; the bottom of the conical section is connected to the first storage tank. The first storage tank is two storage tanks connected in up and down series, and the two storage tanks connected in up and down series may be the same or different. Each of the two storage tanks independently consists of an upper straight cylinder section and a lower conical section, and the taper angle of the lower conical section is 3 to 30°, preferably 5 to 15°. A shut-off valve which is intermittently opened and closed is provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first separator, the inner diameter of the shut-off valve between the upper and lower storage tanks and the interface diameter between the lower storage tank and the first ejector are all 1/8 to 1/4, preferably 1/5 to 1/4 of the diameter of the straight cylinder section of the first separator, and the above three passage diameters may be the same or different.

The top of the lower storage tank of the first storage tank has a pressure equalizing pipeline, and the pressure equalizing pipeline is connected to an atmospheric pressure gas, and the atmospheric pressure gas and the desorption gas are of the same kind. The pressure equalizing pipeline is in gas communication with the lower storage tank. When a negative pressure is generated at the negative pressure inlet of the first ejector, the pressure equalizing gas is pumped into the lower storage tank of the first storage tank to form a downward gas flow, and the desorption gas carrying the adsorbing agent which has adsorbed TBA is introduced into the first ejector and further transported to the second separator.

The second separator consists of an upper straight cylinder section, a middle conical section and a lower second storage tank. The height-to-diameter ratio of the straight cylinder section is 1:1 to 10:1, preferably 2:1 to 4:1; the taper angle of the conical section is 3 to 30°, preferably 5 to 15°; the bottom of the conical section is connected to the second storage tank. The second storage tank is two storage tanks connected in up and down series, and two storage tanks connected in up and down series may be the same or different. Each of the two storage tanks consists of an upper straight cylinder section and a lower conical section, and the lower parts of the upper and lower storage tanks are both in the shape of inverted cones and the taper angle of which is 3 to 30°, preferably 5 to 15°. A shut-off valve which is intermittently opened and closed is provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and

lower storage tanks, and the interface diameter between the lower storage tank and the ejector are $1/8$ to $1/4$, preferably $1/4$ to $1/5$ of the diameter of the straight cylinder section, and the above three passage diameter may be the same or different. Different from the first separator, the inner wall of the second separator has a downward spiral deflector, and the deflector spiral downwards from the connection between the outlet of the first ejector and the second separator and extends along the tangential direction of the inner wall to a position of the second separator where the distance from the position to the bottom of the conical section is $1/3$ to $2/3$ of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section). The deflector can extend the residence time of the solid adsorbing agent in the second centrifugal separator and increase the relative velocity between the driving gas and the solid (adsorbing agent) to improve the desorption efficiency of TBA.

The top of the lower storage tank of the second storage tank also has a pressure equalizing pipeline, and the pressure equalizing pipeline is connected to an atmospheric pressure gas, and the atmospheric pressure gas and the desorption gas are of the same kind. The pressure equalizing pipeline is in gas communication with the lower storage tank. When a negative pressure is generated at the negative pressure inlet of the second ejector, the pressure equalizing gas is pumped into the lower storage tank of the second storage tank to form a downward gas flow, and the downward gas flow is introduced into the second ejector while carrying the adsorbing agent and further transported to the inlet of the suspended bed. Since the adsorbing agent carries a small amount of gas when entering the second ejector and enters the inlet of the suspended bed, the small amount of gas brought into the inlet of the suspended bed also enters the first separator with the liquid-solid two-phase flow at the top of the suspended bed, and the brought small amount of gas forms small bubbles in the liquid, which increases the macroscopic density difference between the liquid and the solid and can improve the separation effect of the first separator.

As shown in FIG. 3, in the method and system of the present invention, the first ejector comprises a driving gas inlet 31, a reducing pipe 32, a throat pipe 33, a negative pressure suction chamber sleeve 34, a negative pressure inlet 35, a mixing pipe 36, a diffusion pipe 37 and an ejector outlet 38, and the negative pressure inlet thereof is connected to the outlet of the lower storage tank of the first storage tank. Since the diameter of the driving gas inlet of the first ejector is larger than the diameter of the throat pipe in the middle of

the ejector (i.e. the smallest diameter), the driving gas velocity at the throat pipe is greater than the velocity at the inlet. According to Bernoulli's equation, in the same flow path, where the velocity is higher, the pressure is lower. That is, when the velocity of the driving gas is sufficiently large, negative pressure can be generated here so that suction effect can be formed at the top inlet (negative pressure inlet).

Preferably, the first ejector is horizontally mounted; between the outlet of the first ejector and the second separator, a horizontal straight pipe, a downwardly inclined straight pipe or a downward spiral pipe, preferably a downward spiral pipe, is provided and has the same diameter as the outlet of the first ejector. In the spiral pipe, the adsorbing agent can be thrown to the wall surface due to the effect of centrifugal force, and since there is friction between the adsorbing agent and the wall surface, the difference between the velocity of the adsorbing agent and the velocity of gas phase becomes larger, thereby facilitating the desorption of TBA in the adsorbing agent.

The second ejector comprises a driving liquid inlet, a reducing pipe, a throat pipe, a negative pressure suction chamber sleeve, a negative pressure inlet, a mixing pipe, a diffusion pipe and an ejector outlet, and the negative pressure inlet thereof is connected to the outlet of the lower storage tank of the second storage tank. The second ejector is horizontally mounted or vertically downwards mounted, preferably vertically downwards mounted, so that the movement direction of the driving liquid and the adsorbing agent is in line with their gravity direction, which enables a greater degree of energy savings.

In the method and system of the present invention, the internal structures of the first ejector and the second ejector are the same and their dimensions may be the same or different. The ejectors include, but are not limited to, various conventional ejectors capable of sucking and transporting material by the negative pressure produced by the acceleration of gas or liquid.

In general, in the first ejector and the second ejector used in the present invention, the diameter D1 of the negative pressure suction chamber sleeve is 1 to 10 times, preferably 2 to 4 times the diameter D4 of the mixing pipe; the diameter D2 of the driving gas/liquid inlet is 0.5 to 4 times, preferably 1 to 2 times the diameter D4 of the mixing pipe; the diameter D3 of the throat pipe is 1/8 to 1/2, preferably 1/4 to 1/3 of the diameter D2 of the driving gas/liquid inlet; The length L1 of the straight pipe section of the throat pipe is 0.2

to 2 times, preferably 0.5 to 1 times the diameter D3 thereof; the 1/2 taper angle A1 of the necking of the throat pipe is 5 to 30°, preferably 10 to 20°; the distance L2 between the outlet of the throat pipe and the inlet of the mixing pipe is 0.5-4 times, preferably 1-2 times the diameter D4 of the mixing pipe; the length L3 of the mixing pipe is 0.5-10 times, preferably 2-6 times D4; the diffusion pipe is a cone with smaller inlet diameter and larger outlet diameter and the 1/2 taper angle A3 thereof is 3 to 15°, preferably 6 to 8°, and the length L4 of the diffusion pipe is 5 to 30 times, preferably 10 to 15 times D4; to avoid breaking the particles due to head-on collision with the side wall, the connection between the mixing pipe and the negative pressure inlet of the suction chamber is also a taper angle structure, and the 1/2 taper angle A2 thereof is 15 to 75°, preferably 30 to 60°.

The material of the first ejector and the second ejector described herein is metal, carbon fiber, glass fiber, engineering plastic, or a composite material of an engineering plastic with one or more of metal, carbon fiber and glass fiber as strength structure. In order to reduce the wear of the adsorbing agent particles, an engineering plastic or a composite material of an engineering plastic with one or more of metal, carbon fiber and glass fiber as strength structure is preferred.

It will be understood by those skilled in the art that the preferred technical features given above can be combined with other technical features to obtain a variety of complete, preferred separation methods or separation systems, as if such preferred separation methods or separation systems are explicitly disclosed herein. As such, it is not necessary to list all the preferred technical solutions. Those skilled in the art can fully know from the above description how to implement a separation method or a separation system containing preferred technical features and obtain corresponding technical effects.

The following examples will further illustrate the content of the present invention, but the present invention is not limited to the listed examples, but also includes any other well-known changes within the scope of the claimed invention.

Example 1:

TBA-DIB azeotrope (from the top of the azeotrope separation tower of the isobutene oligomerization unit) and the adsorbing agent were passed through the second ejector into the suspended bed for adsorption process. The adsorption products (the adsorbing agent

which had adsorbed TBA and material liquid rich in DIB containing a very small amount of TBA) were discharged from the outlet of the suspended bed into the first centrifugal separator for separation. The material liquid rich in DIB containing a very small amount of TBA was discharged from the upper outlet of the first centrifugal separator, and the adsorbing agent which had selectively adsorbed TBA was discharged from the lower outlet of the first centrifugal separator. DIB was directly obtained from the upper outlet of the first centrifugal separator, or after a small amount of TBA-DIB azeotrope had been separated out by distillation, high-purity DIB was obtained. The separated TBA-DIB azeotrope was recycled back to the suspended bed for further separation. The lower outlet of the first storage tank was connected to the first ejector, and the adsorbing agent which had adsorbed TBA and the desorption gas were introduced into the second centrifugal separator through the first ejector. The gas rich in TBA vapor was discharged from the upper outlet of the second centrifugal separator, and the adsorbing agent which had desorbed TBA was discharged from the lower outlet of the second separator. The gas rich in TBA vapor obtained at the upper outlet of the second centrifugal separator was condensed to obtain TBA directly, or the condensed TBA was further distilled to separate out TBA-DIB azeotrope so as to obtain a high-purity TBA, and the TBA-DIB azeotrope was recycled back to the suspended bed for further separation. The lower outlet of the second storage tank was connected to the inlet of the liquid-driven second ejector, and the adsorbing agent after desorption together with TBA-DIB azeotrope was recycled back to the suspended bed through the second ejector for further separation.

The adsorbing agent used was a polystyrene resin loaded with sulfonic acid groups. The loading amount of the sulfonic acid group was 5.5mmol/g. The adsorbing agent had a roundness of 0.001 and an average particle size of 0.1 mm. The adsorbing agent particles with a particle size smaller than 0.7 times the average particle size were less than 5% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 5%. The adsorbing agent had a pore size of 10 nm, a specific surface area of 100 m²/g and a true density of 0.7 g/ml.

The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:1. The linear velocity of the azeotrope in the suspended bed was 20 times the critical suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 1000:1 and was operated at atmospheric pressure and an operating temperature of 25°C.

The pipe provided between the outlet of the suspended bed and the first separator was an equal-diameter straight pipe; the average linear velocity at the inlet of the first centrifugal separator was 50m/s, and the connection between the outlet of the suspended bed and the equal-diameter straight pipe was a 60° taper pipe; the height-to-diameter ratio of the straight cylinder section of the first separator was 1:1, the taper angle of the conical section was 30°, and the bottom of the conical section was connected with the first storage tank. The first storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 30°, and a shut-off valve opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first centrifugal separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all were 1/8 of the diameter of the straight cylinder section of the first centrifugal separator.

The second centrifugal separator had an inlet gas velocity of 50 m/s and an operating temperature of 25°C. The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 1:1, the taper angle of the conical section was 30°, and the bottom of the conical section was connected with the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 30°, and a shut-off valve opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector all were 1/8 of the diameter of the straight cylinder section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector and the second centrifugal separator along the tangential direction of the inner wall to a position of the second centrifugal separator where the distance from the position to the bottom of the conical is 2/3 of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section).

The first ejector was driven by a desorption gas and was horizontally mounted. The pipe

provided between the outlet of the first ejector and the second centrifugal separator was a horizontal straight pipe with the same diameter as the outlet of the first ejector (as shown in FIG. 2). The second ejector was driven by a liquid, the driving liquid was TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank, and the second ejector was horizontally mounted; the driving liquid had a speed of 50m/s at the throat pipe of the second ejector.

The desorption gas was helium, and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage tanks both were connected to helium at atmospheric pressure.

The DIB obtained at the outlet of the first centrifugal separator had a purity of 90 wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope the top of the tower and DIB with a purity of 99wt% from the bottom of the tower. The liquid obtained by condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 81wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99wt% from the bottom of the tower.

Example 2:

Its operation procedure was the same as that of Example 1 and the adopted process flow diagram was shown in FIG. 2. The adsorbing agent used was a polystyrene resin loaded with sulfonic acid group and the polystyrene resin was a copolymer of a copolymer of styrene and divinylbenzene compounded with metal fiber particles. The loading amount of the sulfonic acid group was 3.0 mmol/g. The adsorbing agent had a roundness of 0.1 and an average particle size of 0.5 mm. The adsorbing agent particles with a particle size smaller than 0.7 times the average particle size were less than 4% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 4%. The adsorbing agent had a pore size of 500 nm, a specific surface area of 5 m²/g and a true density of 1.5 g/ml.

The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:10. The linear velocity of the azeotrope in the suspended bed was 1.05 times the critical

suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 1:1 and was operated at atmospheric pressure and an operating temperature of 50°C.

5 The pipe provided between the outlet of the suspended bed and the first separator was a reducing pipe whose taper angle gradually decreased from 60° to 5°, and its minimum cross-sectional area was 20% of its maximum cross-sectional area; the average linear velocity at the inlet of the first separator was 1 m/s, and the connection between the outlet of the suspended bed and the reducing pipe was a round angle and the radius of the round
10 angle was 2 times the maximum diameter of the reducing pipe. The height-to-diameter ratio of the straight cylinder section of the first separator was 10:1, the taper angle of the conical section was 3°, and the bottom of the conical section was connected with the first storage tank. The first storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower
15 conical section with a taper angle of 3°, and a shut-off valve opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all
20 were 1/4 of the diameter of the straight cylinder section of the first separator.

The second centrifugal separator had an inlet gas velocity of 0.5 m/s and an operating temperature of 50°C. The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 10:1, the taper angle of the conical section was 3°, the
25 bottom of the conical section was connected to the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 3°, and a shut-off valve which was opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the
30 bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector all were 1/4 of the diameter of the straight cylinder section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector
35 and the second centrifugal separator along the tangential direction of the inner wall to a

position of the second centrifugal separator where the distance from the position to the bottom of the conical section is $\frac{1}{3}$ of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section).

- 5 The first ejector was driven by a desorption gas and was horizontally mounted. Between the outlet of the first ejector and the second centrifugal separator was a downwardly inclined straight pipe having the same diameter as that of the outlet of the first ejector. The second ejector was driven by a liquid, the driving liquid was TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank and the second ejector was vertically downwards mounted; The driving liquid had a speed of 0.5 m/s at the throat pipe of the second ejector.

15 The desorption gas was carbon dioxide, and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage tanks both were connected to atmospheric pressure carbon dioxide.

The purity of the DIB obtained at the outlet of the first centrifugal separator was 99 wt% and there was no need for the DIB to be purified by distillation. The liquid obtained by condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 89wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20, a feed position at the 9th theoretical plate and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99wt% from the bottom of the tower.

25 **Example 3:**

Its operation procedure was the same as that of Example 1. The adsorbing agent used was a polystyrene resin loaded with sulfonic acid groups and the polystyrene resin was a copolymer of a copolymer of styrene and divinylbenzene compounded with glass fiber particles. The loading amount of the sulfonic acid group was 4.0 mmol/g. The adsorbing agent had a roundness of 0.08 and an average particle size of 0.8 mm. The adsorbing agent particles with a particle size smaller than 0.7 times the average particle size were less than 3% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 3%. The adsorbing agent had a pore size of 300 nm, a specific surface area of 20 m²/g and a true density of 1.2 g/ml.

The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:5. The linear velocity of the azeotrope in the suspended bed was 2 times the critical suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 10:1 and was operated at atmospheric pressure and an operating temperature of 30°C.

The pipe provided between the outlet of the suspended bed and the first separator was a reducing pipe whose taper angle gradually decreased from 50° to 5°, and its minimum cross-sectional area was 30% of its maximum cross-sectional area; the average linear velocity at the inlet of the first centrifugal separator was 3 m/s, and the connection between the outlet of the suspended bed and the reducing pipe was a round angle and the radius of the round angle was 2 times the maximum diameter of the reducing pipe. The height-to-diameter ratio of the straight cylinder section of the first separator was 4:1, the taper angle of the conical section was 5°, and the bottom of the conical section was connected to the first storage tank. The first storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 5°, and a shut-off valve opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first centrifugal separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all were 1/5 of the diameter of the straight cylinder section of the first centrifugal separator.

The second centrifugal separator had an inlet gas velocity of 2 m/s and an operating temperature of 30°C. The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 4:1, the taper angle of the conical section was 5°, and the bottom of the conical section was connected to the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 5°, and a shut-off valve which was opened and closed intermittently was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the

lower storage tank and the ejector all were $1/5$ of the diameter of the straight cylinder section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector and the second centrifugal separator along the tangential direction of the inner wall to a position of the second centrifugal separator where the distance from the position to the bottom of the conical section was $1/3$ of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section).

The first ejector was driven by a desorption gas and was mounted horizontally. The pipe between the outlet of the first ejector and the second centrifugal separator was a spiral downward spiral pipe having the same diameter as that of the outlet of the first ejector; the second ejector was driven by TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank and the second ejector was vertically downwards mounted; The driving liquid had a speed of 2 m/s at the throat pipe of the second ejector. The desorption gas was helium and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage tanks both were connected to atmospheric pressure helium.

The DIB obtained at the outlet of the first centrifugal separator had a purity of 97 wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 0.5, to obtain TBA-DIB azeotrope from the top of the tower and DIB with a purity of 99wt% from the bottom of the tower. The liquid obtained by condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 85wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99wt% from the bottom of the tower.

Example 4:

Its operation procedure was the same as that of Example 1. The adsorbing agent used was a polystyrene resin loaded with sulfonic acid groups and the polystyrene resin was a copolymer of a copolymer of styrene and divinylbenzene compounded with cellulose. The loading amount of the sulfonic acid groups was 5.0 mmol/g. The adsorbing agent had a roundness of 0.05 and an average particle size of 1 mm. The adsorbing agent particles

with a particle size smaller than 0.7 times the average particle size were less than 1% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 1%. The adsorbing agent had a pore size of 200 nm, a specific surface area of 80 m²/g and a true density of 0.8 g/ml.

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The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:2. The linear velocity of the azeotrope in the suspended bed was 5 times the critical suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 40:1 and was operated at atmospheric pressure and an operating temperature of

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The pipe between the outlet of the suspended bed and the first separator was a reducing pipe whose taper angle gradually decreased from 30° to 10°, and its minimum cross-sectional area was 40% of its maximum cross-sectional area; the average linear velocity at the inlet of the first centrifugal separator was 10 m/s, and the connection between the outlet of the suspended bed and the reducing pipe was a round angle and the radius of the round angle was 2 times the maximum diameter of the reducing pipe. The height-to-diameter ratio of the straight cylinder section of the first centrifugal separator was 3:1, the taper angle of the conical section was 10°, and the bottom of the conical section was connected with the first storage tank. The first storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 10°, and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first centrifugal separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all were 1/5 of the diameter of the straight cylinder section of the first centrifugal separator.

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The second centrifugal separator had an inlet gas velocity of 6 m/s and an operating temperature of 40°C. The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 3:1, the taper angle of the conical section was 10°, and the bottom of the conical section was connected to the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper

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angle of 10° , and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector all were $1/5$ of the diameter of the straight cylinder section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector and the second centrifugal separator along the tangential direction of the inner wall to a position of the second centrifugal separator where the distance from the position to the bottom of the conical section is $1/2$ of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section).

The first ejector was driven by a desorption gas and was mounted horizontally. The pipe between the outlet of the first ejector and the second centrifugal separator was a spiral downward spiral pipe having the same diameter as the outlet of the first ejector; the second ejector was driven by TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank and the second ejector was vertically downwards mounted; The driving liquid had a speed of 6 m/s at the throat pipe of the second ejector. The desorption gas was nitrogen and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage tanks both were connected to atmospheric pressure nitrogen.

The DIB obtained at the outlet of the first centrifugal separator had a purity of 94 wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and DIB with a purity of 99% from the bottom of the tower. The liquid obtained by condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 83wt% and was separated in an atmospheric distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99wt% from the bottom of the tower.

Example 5:

Its operation procedure was the same as that of Example 1. The adsorbing agent used was a polystyrene resin loaded with nitro groups and the polystyrene resin was a copolymer of

a copolymer of styrene and divinylbenzene compounded with cellulose. The loading amount of the nitro groups was 1.0 mmol/g. The adsorbing agent had a roundness of 0.05 and an average particle size of 1.5 mm. The adsorbing agent particles with a particle size smaller than 0.7 times the average particle size were less than 1% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 1%. The adsorbing agent had a pore size of 100 nm, a specific surface area of 50 m²/g and a true density of 1 g/ml.

The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:20. The linear velocity of the azeotrope in the suspended bed was 8 times the critical suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 60:1 and was operated at atmospheric pressure and an operating temperature of 30°C.

The pipe between the outlet of the suspended bed and the first separator was a reducing pipe whose taper angle gradually decreased from 30° to 10°, and its minimum cross-sectional area was 50% of its maximum cross-sectional area; the average linear velocity at the inlet of the first centrifugal separator was 20 m/s, and the connection between the outlet of the suspended bed and the reducing pipe was a round angle connection and the radius of the round angle was 2 times the maximum diameter of the reducing pipe. The height-to-diameter ratio of the straight cylinder section of the first centrifugal separator was 3:1, the taper angle of the conical section was 10°, and the bottom of the conical section was connected with the first storage tank. The first storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 10°, and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first centrifugal separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all were 1/5 of the diameter of the straight cylinder section of the first centrifugal separator.

The second centrifugal separator had an inlet gas velocity of 8 m/s and an operating temperature of 30°C. The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 3:1, the taper angle of the conical section was 10°, and

the bottom of the conical section was connected to the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 10° , and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector all were $1/5$ of the diameter of the straight cylinder section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector and the second centrifugal separator along the tangential direction of the inner wall to a position of the second centrifugal separator where the distance from the position to the bottom of the conical section was $1/2$ of the height (based on the height from the top of the second centrifugal separator to the bottom of the conical section).

The first ejector was driven by a desorption gas and was mounted horizontally. The pipe between the outlet of the first ejector and the second centrifugal separator was a spiral downward spiral pipe having the same diameter as that of the outlet of the first ejector; the second ejector was driven by TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank, and the second ejector was vertically downwards mounted; the driving liquid had a speed of 8 m/s at the throat pipe of the second ejector. The desorption gas was nitrogen and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage tanks both were connected to atmospheric pressure nitrogen.

The DIB obtained at the outlet of the first centrifugal separator had a purity of 97 wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 0.5, to obtain TBA-DIB azeotrope from the top of the tower and DIB with a purity of 99wt% from the bottom of the tower. The liquid obtained by condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 8 wt% and was separated in an atmospheric pressure distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99% from the bottom of the tower.

Example 6:

Its operation procedure was the same as that of Example 1 and the adopted process flow diagram was shown in FIG. 1. The adsorbing agent used was a polystyrene resin loaded with amino groups, and the polystyrene resin was a copolymer of a copolymer of styrene and divinylbenzene compounded with cellulose. The loading amount of the amino groups was 10 mmol/g. The adsorbing agent had a roundness of 0.05 and an average particle size of 5 mm. The adsorbing agent particles with a particle size smaller than 0.7 times the average particle size were less than 1% and the adsorbing agent particles with a particle size greater than 1.3 times the average particle size were less than 1%. The adsorbing agent had a pore size of 200 nm, a specific surface area of 50 m²/g and a true density of 1.1 g/ml.

The mass ratio of the TBA in the feed TBA-DIB azeotrope to the adsorbing agent was 1:0.5. The linear velocity of the azeotrope in the suspended bed was 10 times the critical suspending velocity of the adsorbing agent. The suspended bed had a height-to-diameter ratio of 100:1 and was operated at atmospheric pressure and an operating temperature of 60°C.

The pipe between the outlet of the suspended bed and the first separator was a reducing pipe whose taper angle gradually decreased from 25° to 15°, and its minimum cross-sectional area was 80% of its maximum cross-sectional area; the average linear velocity at the inlet of the first centrifugal separator was 30 m/s, and the connection between the outlet of the suspended bed and the reducing pipe was a round angle connection and the radius of the round angle was 2 times the maximum diameter of the reducing pipe. The height-to-diameter ratio of the straight cylinder section of the first centrifugal separator was 2:1, the taper angle of the conical section was 15°, and the bottom of the conical section was connected with the first storage tank. The first storage tank comprised two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper angle of 15°, and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank of the first centrifugal separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and

the interface diameter between the lower storage tank and the first ejector all were $1/5$ of the diameter of the straight cylinder section of the first centrifugal separator.

5 The second centrifugal separator had an inlet gas velocity of 10 m/s and an operating temperature of 60°C . The height-to-diameter ratio of the straight cylinder section of the second centrifugal separator was 2:1, the taper angle of the conical section was 15° , and the bottom of the conical section was connected to the second storage tank. The second storage tank was two storage tanks connected in up and down series, which independently consisted of an upper straight cylinder section and a lower conical section with a taper
10 angle of 15° , and a shut-off valve which was intermittently opened and closed was provided between the upper and lower storage tanks. The passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector all were $1/5$ of the diameter of the straight cylinder
15 section. The downward spiral deflector on the inner wall of the second centrifugal separator spiraled downwards from the connection between the outlet of the first ejector and the second centrifugal separator along the tangential direction of the inner wall to a position of the second centrifugal separator where the distance of the position to the bottom of the conical section was $1/2$ of the height(based on the height from the top of the
20 second centrifugal separator to the bottom of the conical section).

The first ejector was driven by a desorption gas and was mounted horizontally. The pipe between the outlet of the first ejector and the second centrifugal separator was a spiral downward spiral pipe having the same diameter as the outlet of the first ejector; the
25 second ejector was driven by TBA-DIB azeotrope liquid, its negative pressure inlet was connected with the outlet of the lower storage tank of the second storage tank, and the second ejector was mounted horizontally; the driving liquid had a speed of 10 m/s at the throat pipe of the second ejector. The desorption gas was nitrogen and the pressure equalizing pipelines at the top of the lower storage tanks of the first and second storage
30 tanks both were connected to atmospheric pressure nitrogen.

The DIB obtained at the outlet of the first centrifugal separator had a purity of 88 wt% and was separated in an atmospheric distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and
35 DIB with a purity of 99wt% from the bottom of the tower. The liquid obtained by

condensation of the stream from the upper outlet of the second centrifugal separator had a TBA content of 80 wt% and was separated in an atmospheric distillation tower with a theoretical plate number of 20 and a reflux ratio of 1, to obtain TBA-DIB azeotrope from the top of the tower and TBA with a purity of 99 wt% from the bottom of the tower.

CLAIMS

1. A method for separating tert-butanol from diisobutylene, comprising:

(1) passing TBA-DIB azeotrope and an adsorbing agent through a second ejector into a suspended bed to perform an adsorption process, and discharging the adsorption product from the outlet of the suspended bed into a first separator for separation, wherein the obtained material liquid rich in DIB is discharged from the upper outlet of the first separator and the adsorbing agent which has adsorbed the TBA is discharged from the lower outlet of the first separator; preferably, a small amount of TBA-DIB azeotrope is separated out from the material liquid rich in DIB by distillation to obtain high-purity DIB; more preferably, the TBA-DIB azeotrope separated out by distillation is recycled back to the suspended bed; preferably, the mass ratio of the composition of the azeotrope at atmospheric pressure is TBA:DIB=62:38;

(2) introducing the adsorbing agent which has adsorbed TBA and a desorption gas into a second separator through a first ejector for separation, wherein the obtained gas rich in TBA vapor is discharged from the upper outlet of the second separator, and the adsorbing agent which has desorbed TBA is discharged from the lower outlet of the second separator; preferably, the gas rich in TBA vapor is condensed to obtain TBA directly; more preferably, TBA-DIB azeotrope is further separated out by distillation from the TBA obtained by condensation to obtain high-purity TBA; more preferably, the TBA-DIB azeotrope separated out by distillation is recycled back to the suspended bed.

2. The method according to claim 1, further comprising: (3) recycling the adsorbing agent which has desorbed TBA back to the suspended bed through the second ejector.

3. The method according to claim 1 or 2, wherein the adsorbing agent is a polystyrene resin loaded with strong polar groups; preferably, the polystyrene resin is a copolymer of styrene and divinylbenzene and/or a copolymer of a copolymer of styrene and divinylbenzene compounded with short fibers, and the short fibers include, but are not limited to, one or more of metal fiber particles, glass fiber particles and cellulose particles; preferably, the strong polar group is one or more of a sulfonic acid group, a nitro group, an amino group, and a carboxyl group; more preferably, the loading amount of the strong polar group is 0.5 to 10 mmol/g, preferably 3 to 5.5 mmol/g.

4. The method according to claim 3, wherein the adsorbing agent consists of porous spherical particles, and has a roundness of 0 to 0.1, an average particle size of 0.1 to 5 mm, preferably 0.8 to 1.5 mm, and a uniform particle size, based on the total weight of the adsorbing agent, the adsorbing agent particles with a particle size less than 0.7 times the average particle size are not more than 5%, the adsorbing agent particles with a particle size greater than 1.3 times the average particle size are not more than 5%; the pore size of the adsorbing agent is 10-500 nm, preferably 100-300 nm; the specific surface area of the adsorbing agent is 5-100 m²/g, preferably 20-80 m²/g; and the true density of the adsorbing agent is 0.8-1.5 g/ml, preferably 1-1.2 g/ml.

5. The method according to claim 3 or 4, wherein the average length of the short fiber is 1/100-1/2, preferably 1/20-1/5 of the average particle size of the adsorbing agent; the average diameter of the short fiber is 1/10000-1/5, preferably 1/1000-1/10 of the average length.

6. The method according to any one of the preceding claims, wherein the mass ratio of tert-butanol in the TBA-DIB azeotrope to the adsorbing agent at the inlet of the suspended bed is 1:0.5-1:20, preferably 1:2-1:10; preferably, the adsorbing agent is added at one time.

7. The method according to any one of the preceding claims, wherein the TBA-DIB azeotrope and solid adsorbing agent are fed from the inlet at the bottom of the suspended bed; preferably, the feed linear velocity of the TBA-DIB azeotrope is 1.05-20 times, preferably 2-10 times the critical suspension velocity of the adsorbing agent; the average linear velocity of the adsorbing agent in the suspended bed is 1-99%, preferably 50-90% of the linear velocity of the TBA-DIB azeotrope.

8. The method according to any one of the preceding claims, wherein in step (1), the average linear velocity of the adsorbing agent which has adsorbed TBA together with the material liquid rich in DIB entering the first separator from the outlet of the suspended bed is 1-50 m/s, preferably 3-30 m/s; in step (2), the gas velocity at the inlet of the second separator is 0.5-50 m/s, preferably 2-10 m/s and the operating temperature is 25-60°C, preferably 30-50°C.

9. The method according to any one of the preceding claims, wherein the first ejector is driven by a desorption gas and the desorption gas is one or more of nitrogen, carbon dioxide, argon and helium, preferably nitrogen and/or carbon dioxide; the temperature of the desorption gas of the first ejector is 25-60°C, preferably 30-50°C;

5 the second ejector is driven by a liquid and the driving liquid is TBA-DIB azeotrope liquid; the driving liquid has a maximum velocity at the throat pipe of the second ejector of 0.5-50 m/s, preferably 2-10m/s.

10. The method according to any one of the preceding claims, wherein the operating pressure of the suspended bed is atmospheric pressure; the operating temperature is between 25°C and the boiling point of TBA-DIB azeotrope.

11. A system for separating tert-butanol from diisobutylene, comprising:

a suspended bed, wherein the feed pipe of the suspended bed is connected to a DIB-TBA azeotrope supply pipe through a second ejector, and the outlet of the suspended bed is connected to the inlet of a first separator;

a first separator, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the suspended bed, the upper outlet is used for discharging material liquid rich in DIB, and the lower outlet is connected to a second separator through a first ejector;

a second separator, comprising an inlet, an upper outlet and a lower outlet, wherein the inlet is connected to the first separator through the first ejector, the upper outlet is used for discharging gas rich in TBA vapor, and the lower outlet is used for discharging adsorbing agent which has desorbed TBA; preferably, the lower outlet of the second separator is connected to the feed pipe of the suspended bed through the second ejector.

12. The system according to claim 11, wherein the upper outlet of the first separator is connected to a first distillation tower through a pipe; the upper outlet of the first distillation tower is used for discharging TBA-DIB azeotrope, and the lower outlet of the first distillation tower is used for discharging high-purity DIB; preferably, the upper outlet of the first distillation tower is connected to the TBA-DIB azeotrope supply pipe.

13. The system according to claim 11 or 12, wherein the upper outlet of the second separator is connected to a condenser through a pipe, or the upper outlet of the second separator is connected to a condenser through a pipe and the material outlet of the

condenser is connected to the inlet of a second distillation tower; preferably, the upper outlet of the second distillation tower is used for discharging TBA-DIB azeotrope, and the lower outlet of the second distillation tower is used for discharging high-purity TBA; preferably, the upper outlet of the second distillation tower is connected to the TBA-DIB azeotrope supply pipe.

14. The system according to any one of claims 11-13, wherein the height-to-diameter ratio of the suspended bed is 1000:1-1:1, preferably 100:1-10:1; preferably, the inlet at the bottom of the suspended bed is an equal-diameter straight pipe, a reducing pipe or a combination thereof, preferably a reducing pipe whose taper angle gradually decreases, and the taper angle is between 0 and 60°, preferably between 0 and 30°; the minimum cross-sectional area of the passage of the reducing pipe is 20-80%, preferably 30-50% of the maximum cross-sectional area of the passage of the reducing pipe; preferably the outlet of the suspended bed is a round angle outlet or a taper angle outlet.

15. The system according to any one of claims 11-14, wherein the outlet of the suspended bed and the inlet of the first separator are connected by an equal-diameter straight pipe or a reducing pipe or a combination thereof, preferably a reducing pipe whose taper angle gradually decreases, wherein the maximum taper angle does not exceed 60°, the minimum taper angle is not less than 5°, preferably between 10 and 30°; preferably, the minimum cross-sectional area of the reducing tube is 20-80%, preferably 30-50% of the maximum cross-sectional area of the reducing tube.

16. The system according to any one of claims 11-15, wherein the first separator and the second separator are centrifugal separators.

17. The system according to claim 16, wherein the first separator consists of an upper straight cylinder section, a middle conical section and a lower first storage tank; the height-to-diameter ratio of the straight cylinder section is 1:1-10:1, preferably 2:1-4:1; the taper angle of the conical section is 3-30°, preferably 5-15°; the bottom of the conical section is connected to the first storage tank; preferably, the first storage tank is two storage tanks connected in up and down series and each of them independently consists of an upper straight cylinder section and a lower conical section, and the taper angle of the lower conical section is 3-30°, preferably 5-15°; preferably, a shut-off valve which is intermittently opened and closed is provided between the upper and lower storage tanks;

the passage diameter between the bottom of the conical section and the upper storage tank of the first separator, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the first ejector all are $1/8$ - $1/4$, preferably $1/5$ - $1/4$ of the diameter of the straight cylinder section of the first separator; preferably, the top of the lower storage tank of the first storage tank has a pressure equalizing pipeline, and the pressure equalizing pipeline is connected to an atmospheric pressure gas and in gas communication with the lower storage tank.

18. The system according to claim 16, wherein the second separator consists of an upper straight cylinder section, a middle conical section and a lower second storage tank; the height-to-diameter ratio of the straight cylinder section is $1:1$ - $10:1$, preferably $2:1$ - $4:1$; the taper angle of the conical section is 3 - 30° , preferably 5 - 15° ; the bottom of the conical section is connected to the second storage tank; preferably, the second storage tank is two storage tanks connected in up and down series and each of them consists of an upper straight cylinder section and a lower conical section, and the lower parts of the upper and lower storage tanks are both in the shape of inverted cones with an taper angle of 3 - 30° , preferably 5 - 15° ; preferably, a shut-off valve which is intermittently opened and closed is provided between the upper and lower storage tanks; the passage diameter between the bottom of the conical section and the upper storage tank, the inner diameter of the shut-off valve between the upper and lower storage tanks, and the interface diameter between the lower storage tank and the ejector are $1/8$ - $1/4$, preferably $1/4$ - $1/5$ of the diameter of the straight cylinder section; preferably, the inner wall of the second separator has a downward spiral deflector, and the deflector spirals downwards from the connection between the outlet of the first ejector and the second separator and extends along the tangential direction of the inner wall to a position of the second separator where the height from the position to the bottom of the conical section is $1/3$ - $2/3$ of the height from the top of the second centrifugal separator to the bottom of the conical section; preferably, the top of the lower storage tank of the second storage tank also has a pressure equalizing pipeline, and the pressure equalizing pipeline is connected to an atmospheric pressure gas and in gas communication with the lower storage tank.

19. The system according to any one of claims 11-18, wherein the first ejector comprises a driving gas inlet, a reducing pipe, a throat pipe, a negative pressure suction chamber sleeve, a negative pressure inlet, a mixing pipe, a diffusion pipe and an ejector outlet, wherein the negative pressure inlet is connected to the outlet of the lower storage

tank of the first storage tank; preferably, the first ejector is horizontally mounted; between the outlet of the first ejector and the second separator, a horizontal straight pipe, a downwardly inclined straight pipe or a downward spiral pipe is provided and has the same diameter as the outlet of the first ejector.

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20. The system according to any one of claims 11-19, wherein the second ejector comprises a driving liquid inlet, a reducing pipe, a throat pipe, a negative pressure suction chamber sleeve, a negative pressure inlet, a mixing pipe, a diffusion pipe and an ejector outlet, wherein the negative pressure inlet is connected to the outlet of the lower storage tank of the second storage tank; preferably, the second ejector is horizontally mounted or vertically downwards mounted.

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21. The system according to any one of claims 19-20, wherein in the first ejector and the second ejector, the diameter of the negative pressure suction chamber sleeve is 1-10 times, preferably 2-4 times the diameter of the mixing pipe; the diameter of the driving gas or driving liquid inlet is 0.5-4 times, preferably 1-2 times the diameter of the mixing pipe; the diameter of the throat pipe is $1/8-1/2$, preferably $1/4-1/3$ of the diameter of the driving gas/liquid inlet; The length of the straight pipe section of the throat pipe is 0.2-times, preferably 0.5-1 times the diameter thereof; the $1/2$ taper angle of the necking of the throat pipe is $5-30^\circ$, preferably $10-20^\circ$; the distance between the outlet of the throat pipe and the inlet of the mixing pipe is 0.5-4 times, preferably 1-2 times the diameter of the mixing pipe; the length of the mixing pipe is 0.5-10 times, preferably 2-6 times the diameter of the mixing pipe; the diffusion pipe is a cone with smaller inlet diameter and larger outlet diameter and the $1/2$ taper angle thereof is $3-15^\circ$, preferably $6-8^\circ$, and the length of the diffusion pipe is 5-30 times, preferably 10-15 times the diameter of the mixing pipe; the connection between the mixing pipe and the negative pressure inlet of the suction chamber is a taper angle structure, and the $1/2$ taper angle A_2 thereof is $15-75^\circ$, preferably $30-60^\circ$.

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22. The system according to any one of claims 11-21, wherein the material of the first ejector and/or the second ejector is metal, carbon fiber, glass fiber, engineering plastic, or a composite material of an engineering plastic with one or more of metal, carbon fiber and glass fiber as strength structure.

ABSTRACT**METHOD AND SYSTEM FOR SEPARATING T-BUTANOL FROM DIISOBUTYLENE**

- 5 Provided are a method and system for separating a t-butanol and diisobutylene azeotrope. The purpose of separating t-butanol from diisobutylene is achieved by allowing the selective adsorption of t-butanol with an adsorbing agent flowing together with t-butanol and diisobutylene in a suspended bed, and performing continuous separation of diisobutylene from the adsorbing agent and continuous regeneration of the adsorbing agent using devices such as separator and ejector. In the method, no extraction agent is introduced, no large-scale, high-pressure equipment is used, the operating conditions are mild, the energy consumption is low and the safety risk is low.
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