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Simulation and Experimental Research of Cyclohexane Isopropyl Alcohol Azeotropic Solvent Separation During Chemical and Pharmaceutical Process

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In this paper, a cyclohexane-isopropanol azeotrope separation process was studied, respectively general simulation the separation process. The steady-state pressure swing distillation process, while at the laboratory of transformer rectification section, reduction pressure distillation column and intermittent small scale experimental atmospheric tower portion. Salt pressure swing distillation experiments, have achieved good results. This article will use dynamic simulation of distillation processes to separate these two processes in line, especially in heterogeneous batch azeotropic distillation and batch extractive distillation, distillation is an attempt to design and method of open up. The general process for separating a cyclohexane-isopropanol azeotrope was simulated optimization studies, including a total theoretical plate number of each section of the tower. Each column operating parameters have been best products of cyclohexane and isopropanol concentrations were reached more than 99.9wt%.

1. Introduction

Chemical separation technology is an important branch of chemical engineering, any chemical processes are inseparable from this technology (Li and An, 2015). A typical chemical process, from refining raw materials, intermediate product separation, purification and waste water, waste gas treatment products are dependent on chemical separation technology (Balog, 2007). Materials and reaction products of the vast majority of the obtained reaction mixture, the system needs to use the differences in the physical properties of the components by means of a separating agent or a separating and purifying the resulting mixture (Choi, 2016).
Since the application areas of chemical separation technology is very broad, raw materials, products and separating operation of a variety of requirements, which determines the diversity of separation technology (Li and Haghtalab, 2015). By dividing mechanism can be roughly divided into five categories, namely: to create a new phase separation (such as distillation, crystallization); adding a new phase separation (such as extraction, absorption); separation (membrane separation) with a spacer (Sawamura, 2015). Solid separation reagent (such as adsorption, ion exchange) and separation (centrifugal separation extraction, electrophoresis) with a force field or gradient, their characteristics and design methods is different (Park, 2016).
In this paper, a method of separating cyclohexane-isopropyl azeotrope, including literature existing methods and transformer rectifier transformer salt and a method of distillation, the use of chemical process simulation software stability of the two methods state process simulation, test the total number of theoretical plates, the impact of feed temperature, feed raw materials and recycling location, reflux ratio and other process parameters on the separation process, and then analyse the optimum process operating parameters sensitivity; for transformer vacuum tower distillation process and the high-pressure column are intermittent experimental study, in which vacuum distillation tower also conducted experiments with salt. Comparing simulation results with experimental data summarize the advantages and disadvantages of various methods of separation. And conduct research for insufficient pressure swing distillation, rectification transformer propose energy-saving measures. The use of salt to improve the rectification cycle pressure swing distillation capacity, high energy deficiencies.

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2. The related theory and method

2.1 The basic principle of rectification

Distillation process principles can be used t-x-y system diagram shown in Figure 1 was explained. The composition of the mixture was raised to xF bubble point to partially vaporized, and the vapor and liquid phases separate to form two phases are y1 and x1, this time y1> xF> x1, vapor-liquid two-phase flow by a lever determination rule (Sheikholeslamzadeh and Buonomenna, 2015). If a composition of x1 continued partially vaporized liquid, the composition can be obtained, respectively y2 'and x2' of the vapor and liquid (not shown), so many times the partially vaporized liquid mixture, the liquid phase and the less volatile component can be obtained with high purity. Meanwhile, the composition of partially condensed vapor phase mixture of y1, y2 can be obtained for the composition of the vapor and the composition of the liquid phase of x2. Continue to be composed of y2 partially condensed vapor phase can be obtained y2 composition of the vapor and liquid phase composition x3, apparently y3> y2> y1. Thus, after repeated vapor mixture partially condensed volatile components in the vapor phase can be obtained with high purity (Basile and Minelli, 2015).

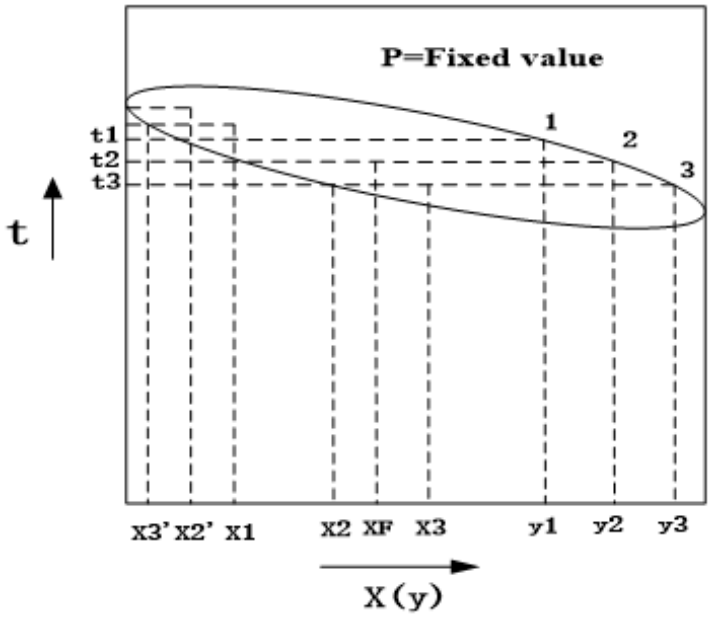

Figure 1: Several partial vaporization and condensation of t-x-y

Above were partially vaporized liquid and vapor phases several times partial condensation process, the two components can be obtained on the principle of separation of high purity, but because a large amount of product obtained middle distillate and leaving less, the yield is very low, and large equipment.

2.2 Intermittent constant reflux ratio of distillation (Micovic, 2014)

In constant reflux ratio of batch distillation process, the kettle liquid composition and distillate composition xW, xD and lower, so the operating initial distillate composition must be higher than the average composition to ensure that the average composition of the distillate meet the quality requirements (Vithanage, 2015). Typically, when the kettle liquid composition reaches a predetermined value, it stops precise operation (Szekely, 2014).

Constant reflux ratio of batch distillation, distillate composition and kettle liquid composition having a correspondence relationship calculation to operate the initial state as a reference, then the kettle liquid composition xD1 (this value is higher than the average distillate composition by design who assumed). According to the minimum reflux ratio is defined by xD1, xF and vapor-liquid equilibrium relationship can be obtained Rmin:

$$R_{\min} = \frac{x_{D1} - y_F}{y_F - x_F} \tag{1}$$

Constant reflux ratio of batch distillation, xD (or xW) and W, D relationship between the differential mass balance should be obtained through:

$$\ln \frac{F}{W_e} = \int_{x_{W_g}}^{x_F} \frac{dx_w}{x_D - x_W} \tag{2}$$

Wherein the amount of bottoms W-- and kettle liquid composition xWe corresponding, kmol.
When a batch operation batch distillation material constant calculation and continuous distillation similar to that:
Total material balance:

$$D = F - W \tag{3}$$

Constant volatile component count:

$$Dx_{Dm} = Fx_F - Wx_W \tag{4}$$

Simultaneous two equations, the solution was:

$$x_{Dm} = \frac{Fx_f - Wx_W}{F - W} \tag{5}$$

The distillate composition xD and eventually bottoms composition xWe, the following formula for the Minimum reflux ratio, that is

$$R_{\min} = \frac{x_D - y_{We}}{y_{We} - x_{We}} \tag{6}$$

2.3 Cyclohexane - isopropyl separation methods

Close to the boiling point of isopropanol and cyclohexane, and the formation of an azeotrope cannot be isolated directly by simple distillation. Since both water and isopropanol hydroxy, easy to form hydrogen bonds with each other, can be miscible in any proportion, cyclohexane and water-immiscible. Accordingly, the first water as extraction agent, isopropanol and cyclohexane separation system is divided into the oil phase (containing mainly cyclohexane) and water phase (mainly consisting of isopropyl alcohol and water) two phases were then separated and purified. The oil phase is rectified to give the desired product can meet the purity requirements of cyclohexane; the aqueous phase by distillation particular, can obtain high purity isopropyl alcohol. General separation process was shown in Figure 2:

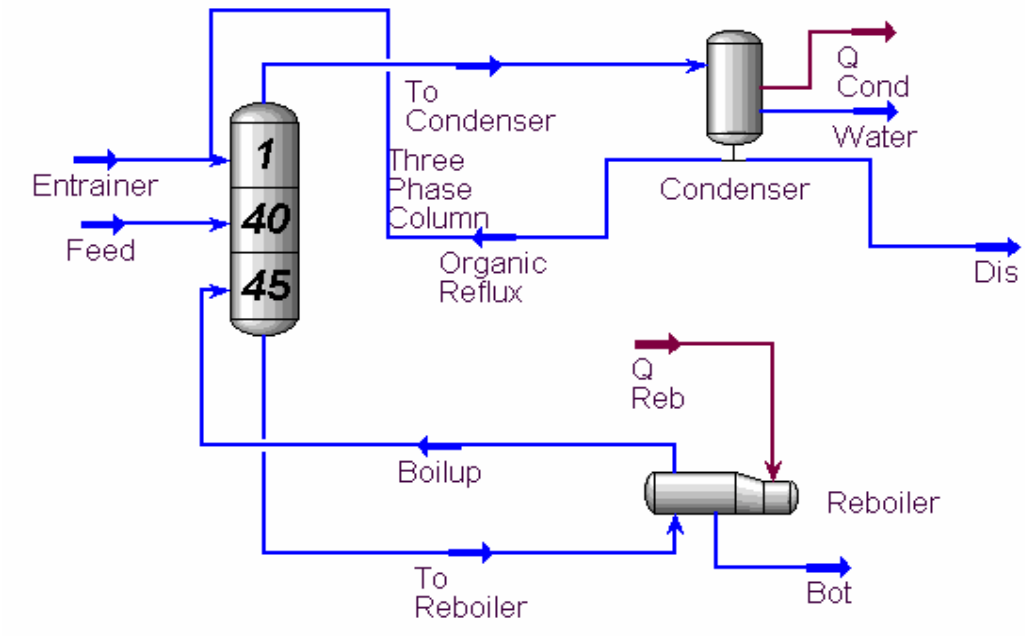

Figure 2: Flow separation method schematic

After changing the heat treatment temperature and pressure H1, the FEED cyclohexane purifying column feed, feed rate of 1000kg / h, where cyclohexane is 91.07wt%, isopropanol 8.93wt%, the central tower into the cyclohexane refining column T1. The fixed feed conditions, respectively, the total number of theoretical plates tower T1, reflux ratio, feed location and the effect of feed temperature on product quality analysis.

3. Experiments and results

3.1 Setting parameters and initial conditions

Structural and operational parameters are listed in Table 1. 10^4

Table1: Technical features and operating conditions of the column

parameter	value	parameter	value
Cross section area[m^2]	0.6567	Equal plate height[m]	0.4965
The diameter of the column[m]	0.9144	Tower height[m]	22.34
Heat load of the boiler[kJ/h]	3.909×10^4	Flow rate of liquid product[kg/h]	1000
condenser heat load[kJ/h]	-3.653×10^4	Produced gas flow[kg/h]	0
Condenser volume[m^3]	4.5	Volume of re boiling device[m^3]	10

Simulation of the initial conditions was as follows:
(1) Distillation column consisting of 45 theoretical plates, respectively, a tower reactor overhead condenser.
(2) By a PID controller by adjusting the back flow control condenser level setting is 40%.
(3) The simulation does not take full reflux stage, but from the beginning of product recovery calculation, the tower was held at the beginning of the steady-state condition by the simulation results given.
(4) Since the total reflux process can be mined portion of the water from the phase separator, so that the simulation does not consider this part of the water. The water content of raw materials is less than the real time simulation, the water: cyclohexane (molar ratio) = 0.5467: 0.1563: 0.2970.
(5) The initial level condenser and reboiler were 0% and 60%.

3.2 Affect the total number of theoretical plates

Other operating parameters fixed column, affect the total number of theoretical plates of the column separation performance was shown in Figure 3: With the increase in the total number of theoretical plates, bottoms remaining isopropyl alcohol content lower bottoms energy consumption decreased slightly. However, when the total number of theoretical plates reached 18, bottoms remaining isopropanol concentration is close to zero, continue to increase the total number of theoretical plates, one-time investment costs increased as the tower slowly The product concentrations. In the late bottoms energy remained unchanged. So choose the total number of theoretical plates is 18.

3.3 Impact of reflux

The remaining concentration of isopropyl alcohol in the bottoms reflux ratio increases and decreases when the reflux ratio was increased to 1.7, reduce the magnitude of decrease bottoms remaining: Figure 4 reflux ratio on the separation performance of the column as shown already down to a lower concentration of isopropyl alcohol. With the increase reflux ratio bottoms consumption soared, due reflux ratio increases, the return of reflux liquid column increased column load increases. So choose a reflux ratio of 1.7.
After the first extraction, the mixture was separated into oil and water phases of two parts, the oil phase is chiefly cyclohexane and minor amounts of isopropyl alcohol, cyclohexane by refining column, the column reactor to close a cyclohexane purity; the aqueous phase in a mixture of isopropanol and water at atmospheric pressure, isopropyl alcohol and water form an azeotrope using extractive distillation to separate the twin towers by continuous operation, in extractive distillation column overhead to give a concentration of 99. 96wt% of isopropyl alcohol, water and bottoms extractant glycol mixture, recovered by solvent recovery column, after recovery, the tower reactor to obtain a concentration of 99.90wt% ethylene glycol can be recycled.

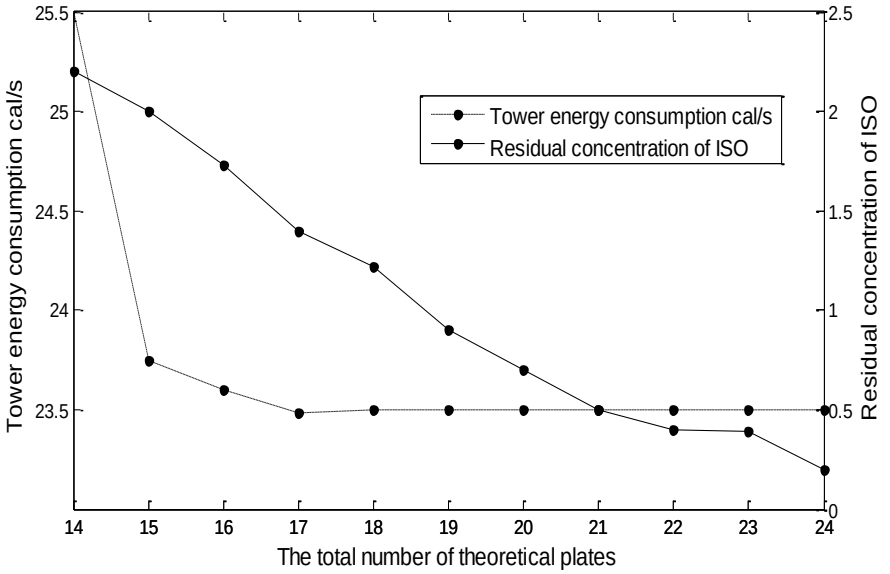

Figure 3: Affect the total number of theoretical plates of the column process indicators

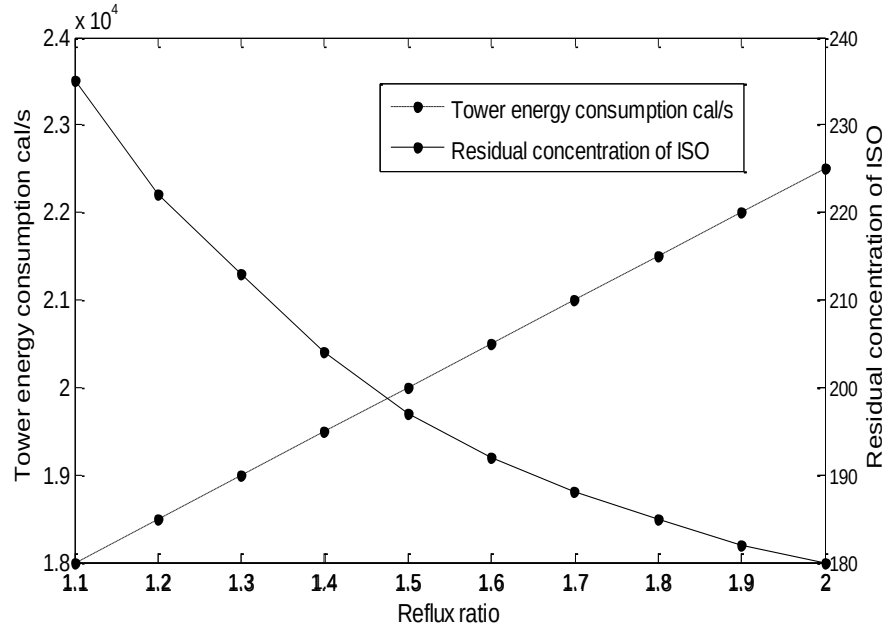

Figure 4: Reflux ratio of column process indicators

4. Conclusions

In this paper, a method of separating cyclohexane-isopropyl azeotrope, including literature existing methods and transformer rectifier transformer salt and a method of distillation, the use of chemical process simulation software stability of the two methods state process simulation, test the total number of theoretical plates, the impact of feed temperature, feed raw materials and recycling location, reflux ratio and other process parameters on the separation process, and then analyse the optimum process operating parameters sensitivity; for transformer vacuum tower distillation process and the high-pressure column are intermittent experimental study, in which vacuum distillation tower also conducted experiments with salt. This article will use dynamic simulation of distillation processes to separate these two processes were in line, especially in

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