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# Dynamic-accumulative operation policy of continuous distillation for the purification of anisole

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In the B10 isotope enrichment industry, the purification of anisole mixture makes great sense. A dynamic-accumulative operation policy of continuous distillation (DACD) with repeated filling and dumping of the still is proposed for the separation of trace heavy impurities in the recycled anisole. To simulate and optimize the purification process of anisole, a mathematical model of DACD is derived, and the computer codes are developed in the MATLAB environment. Moreover, the experiment is performed in a pilot-scale distillation column. The results show that the experimental date agrees well with simulation results. DACD could solve the difficulty of flow rate control when the bottom flow rate is very small in continuous distillation. The size of the still in this operation mode is also smaller than that in batch distillation. And the yield of anisole is raised to 99.91%. In a word, DACD is especially suitable for separating trace heavy impurities from the recycled anisole.

**Keywords:** anisole recycling, dynamic-accumulative operation policy, continuous distillation, simulation.

## INTRODUCTION

Naturally occurring boron has two stable isotopes: $B^{10}$ and $B^{11}$ with the abundances of 19.8% and 80.2% respectively[1]. The impetus for the isotopic fractionation of boron arises primarily from the contrasting difference in thermal neutral capture cross sections. $B^{10}$ has a thermal neutral capture cross section of 3837 barns, while that of $B^{11}$ is only 0.005 barns. It is precisely this propensity for thermal neutron capture which makes $B^{10}$ enriched materials become a useful commodity[2–3]. $B^{10}$ is widely used in nuclear industry[4], defense industry, medicine, and so on[5]. So far, many kinds of methods for $B^{10}$ isotope enrichment have been developed such as chemical exchange rectification of $BF_3$, low temperature rectification of $BF_3$, ion exchange of boric acid, and counter-current circulation of $BF_3$ in the series film[6–12]. Nowadays chemical exchange rectification of boron trifluoride-anisole complex is considered to be the most practical method in the $B^{10}$ isotope enrichment industry[13–14]. In the exchange system of boron trifluoride-anisole, anisole is recycled to the recombiner by the pyrolysis of $CH_3OC_6H_5 \cdot BF_3$ complexes[15]. However, some undesirable side effects come out under the pyrolysis temperature and by-products such as phenol, alkyl anisole and other impurities are formed. In order to reduce the adverse impact of impurities on the concentration of $B^{10}$, the anisole used as complexing agent should contain no more than 1% methylanisole and 0.2–0.3% phenols[16].

The purification of anisole by batch distillation and continuous distillation have been studied. However, the yield of anisole is only 90.46% and 98.64% respectively[17–18]. In addition, ordinary distillation process, either batch or continuous is not suitable for the case where heavy impurities is in very low concentration and at the same time the yield of anisole should be increased as high as possible. In such a case, the amount of the heavy impurities at the bottom of column is so small compared to that of feed, which it is difficult to form practically a stream, or even cannot be separated from a continuous or batch distillation column of practical size[19–20].

The objective of the current work is to study the dynamic-accumulative operation policy of continuous distillation (DACD) for the purification process of anisole mixture. In Section 2, the mathematical model of DACD is derived, and the computer codes are developed in the MATLAB environment to simulate the purification process. The operating parameters are determined by optimizing the initial reboiler holdup and the time to start the second period in Section 3. In Section 4, the design of a pilot-scale distillation column is carried out, the practical implementation of DACD and the experimental results are discussed in details. Some concluding remarks are made on DACD in purifying anisole in the last section of this article.

## THE PROCESS AND THE DYNAMIC MODEL

### The process

The dynamic-accumulative operation policy of continuous distillation (DACD) which illustrated in Figure 1 is characterized by repeating three periods of operation: 1) At the beginning of the operation, the still pot is filled with a specified amount of feed charge. The continuous distillation operation starts and goes on until the purity of the distillate decreases to 0.997 or the specification is satisfied for the residue in the reboiler. During this period, the distillate flow rate is equal to the feed flow rate, so heavy component is enriched in the bottom gradually. 2) Keep all the conditions same, at the same moment the liquid in the reboiler is partially drained out. The drain time is determined by the amount of the removal and the average flow rate of the bottom stream. Note that at the end of the second period the amount of the liquid in the bottom should be sufficient to make the vapor flow rate from the reboiler remain stable during the whole process. 3) In the third period the column is run under total reflux and the liquid holdup of the bottom is accumulated. The continuous distillation operation (the second cycle) begins after accumulated liquid in the reboiler reaches the initial amount. By above mode, the

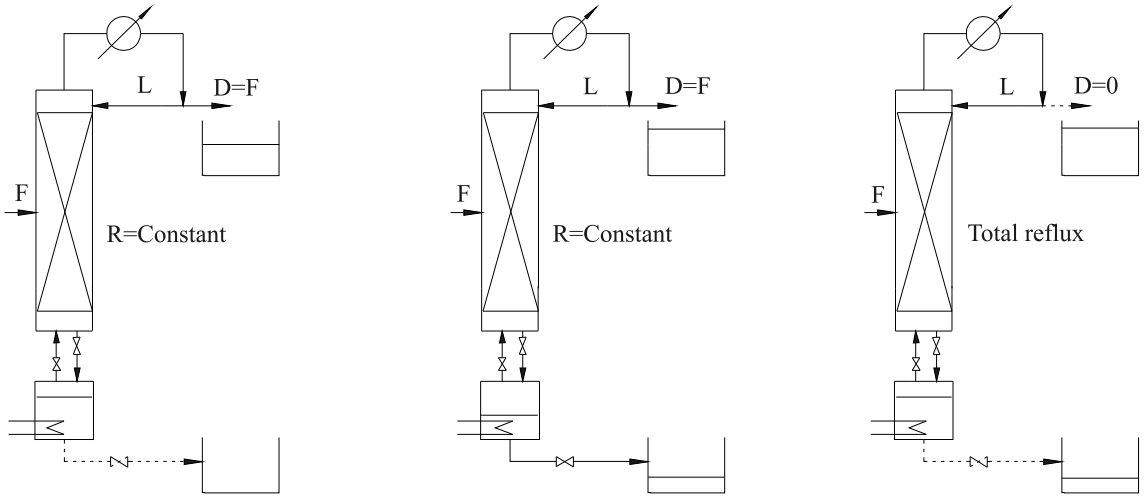


**Figure 1.** The three characteristic periods in the dynamic accumulation operation of continuous distillation

three steps procedure is repeated during the purification process of anisole.

**The dynamic mode of DACD**

For the simulation of the dynamic-accumulative operation policy for continuous distillation (DACD), a mathematical model is presented. The following simplifying assumptions are applied:

– Theoretical trays;
– Constant molar flow;
– Constant molar liquid holdups on theoretical trays;
– Perfect mixing on all trays and in the reboiler;
– Total condenser.

Material balance equations:

1) For the rectifying section (j = 1, 2 …$N_f$ –1)

$$M\frac{dx_j}{dt} = L\left(x_{j-1} - x_j\right) + V\left(y_{j+1} - y_j\right) \tag{1}$$

2) For the feeding plate (j = $N_f$)

$$M\frac{dx_j}{dt} = Lx_{j-1} - L'x_j + V'y_{j+1} - Vy_j + Fz_j \tag{2}$$

3) For the stripping section (j = $N_f$ +1, $N_f$ +2 … N–1)

$$M\frac{dx_j}{dt} = L'\left(x_{j-1} - x_j\right) + V'\left(y_{j+1} - y_j\right) \tag{3}$$

4) For the reboiler (j = N)

In the first period:

$$M_B\frac{dx_N}{dt} = L'x_{N-1} - V'y_N \tag{4}$$

Among the equation, $M_B$ means the initial reboiler holdup (kg).

In the second period:

$$\frac{d\left[(M_B - Wt)x_B\right]}{dt} = L'x_{N-1} - V'y_N - Wx_B \tag{5}$$

Among the equation, $W$ means flow rate of the bottom stream in the second period (kg $h^{-1}$), $x_B$ means liquid composition in the still, and $t$ means the drain time in the second period (h).

In the last period:

$$\frac{d\left(M''_B x_B\right)}{dt} = L'x_{N-1} - V'y_N \tag{6}$$

$$M''_B = \omega + \left(L' - V'\right)t \tag{7}$$

Among the equations, $M_B''$ means reboiler instantaneous holdup in the third period (kg), and ω means reboiler holdup at the end of the second period (kg).

5) Vapor-liquid equation:

$$y_j = \frac{\alpha x_j}{1 + (\alpha - 1)x_j} \tag{8}$$

6) Component addition equation:

$$\sum_{j=1}^{N} x_{j,i} - 1 = \sum_{j=1}^{N} y_{j,i} - 1 = 0 \tag{9}$$

The computer codes are developed in the MATLAB environment to simulate the process and investigate the influence of different parameters. Ode113 is used to solve differential equations and get the concentration parameters on each tray. The change of the concentration in top and bottom over time during the whole purification process of anisole is showed below.

**Model parameters**

The anisole mixture used in this study is produced in the pilot-plant of $B^{10}$ enrichment experiments. It is made up of 99 wt% of anisole, 0.2 wt% of 4-methylanisole, 0.2 wt% of 2-methylanisole and 0.6 wt% of phenol. The relative volatilities of anisole and heavy impurities are 2.30, 1.78 and 1.66 for phenol, 4-methylanisole and 2-methylanisole respectively. There is no azeotropic phenomenon between anisole and any one of the three kinds of heavy impurities. In order to turn multicomponent system to binary system, which simplifies the calculations and makes the simulation in the MATLAB environment easier to carry on, this paper assumes that the heavy impurity is 2-methylanisole alone (the relative volatility is the lowest). As is known to all, the smaller the relative volatility, the harder the separation difficulty. If the binary separation system of anisole and 2-methylanisole can be realized effectively, the multicomponent system of anisole and all the heavy impurities can be realized more easily. So this paper assumes the feed is consisted of 99 wt% of anisole and 1 wt% of 2-methylanisole. During the process of $B^{10}$ enrichment, in order to reduce the adverse impact of impurities, the anisole used as complexing agent should contain no more than 1 wt% methylanisole and 0.2 wt%–0.3 wt% phenols[13]. Therefore, in this paper, the specified product composition is 99.7 wt% of anisole.

**Table 1.** Specification for the computation

| | | | |
|---|---|---|---|
| Feed flow rate [kg $h^{-1}$] | 10 | Feed composition | 0.99 |
| Feed state | q = 1 | Relative volatility | 1.66 |
| Initial reboiler holdup [kg] | 10 | Tray holdup [kg] | 0.25 |
| Condenser holdup [kg] | 1 | Reflux ratio | 1 |
| Vapor flow rate in the rectifying section [kg $h^{-1}$] | 20 | Number of theoretical plates | 35 |
| Reboiler holdup at the end of the second period [kg] | 2 | The position of the feeding theoretical plate | 16 |
| Specified product composition | 0.997 | Flow rate of the bottom stream in the second period [kg $h^{-1}$] | 10 |
| The condition to start the second period | xD = 0.997 | Reflux ratio in the third period | 1E10 |

Fenske equation is used to calculate the minimum number of theoretical plates, Underwood equation calculates the minimum reflux ratio, Gilliland equation calculates the number of theoretical plates and the position of the feeding plate is calculated by Graphical method. The calculated minimum reflux ratio is 0.71. Considering the operational cost and equipment cost, value of 1 is taken as the practical reflux ratio which is 1.3 times as large as the minimum reflux ratio. The specifications are summarized in Table 1. Unless otherwise mentioned, all concentration in this paper should be interpreted to be the concentration of anisole.

**Simulation results and discussions**

The dynamic action of dynamic accumulation continuous distillation column is simulated and the concentrations as a function of time are given in Figure 2 and Figure 3.

Compared to the first period, the second period and the third period take very short time. In order to indicate

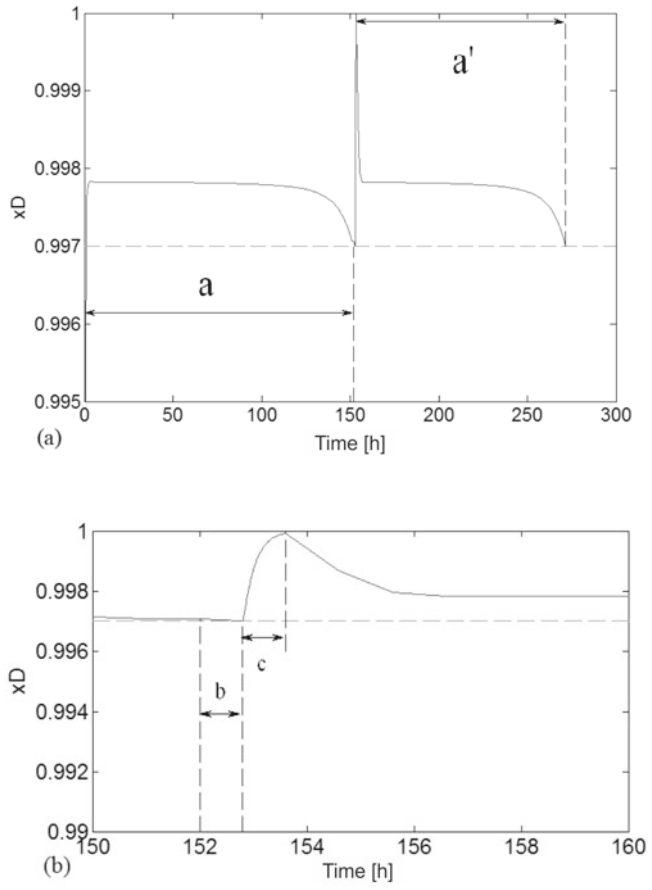


**Figure 2.** Dynamic curve of concentration in top
(a) Overall chart a: the first period in Circle I; a': the first period in Circle II; (b) local chart b: the second period; c: the third period

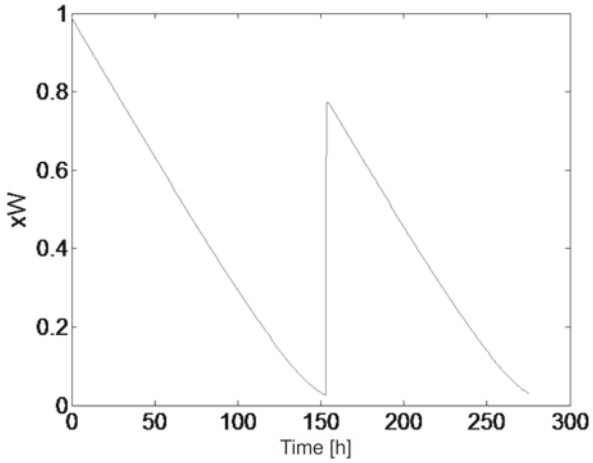


**Figure 3.** Dynamic curve of concentration in bottom

all periods clearly, overall chart and local chart are put to use. Figure 2(a) shows the overall chart containing the first period in Circle I (start-up period), the second period, the third period and the first period in Circle II. Figure 2(b) shows the local chart containing the second period and the third period. In these Figures, a, b, c, and a' represent the first period in Circle I, the second period, the third period and the first period in Circle II, respectively. As shown in Figure 2 and Figure 3, the top concentration increases to 0.9975 rapidly and decreases slowly. After 152 hours, the top concentration decreases to 0.997 while the bottom concentration decreases to 0.0296. That's the first period in Circle I. In the second period, the reboiler holdup is drained out partially and the concentration in top remains at 0.997 owing to a little time consumption on the second period (48 minutes). In the third period, the concentration in top and bottom increases to 0.9998 and 0.7729 respectively within 48 minutes. At the end of the first period in Circle II, the top concentration decreases to 0.997 again and the second period and third period in Circle II is the same as that in Circle I. The following circles repeat the three periods in Circle II. It takes 153.6 hours to finish the start-up circle (Circle I), while when the dynamic accumulation of continuous distillation is relatively stable in the repetition of the three-steps operation, it takes 119.7 hours to finish one circle (Circle II and the followings).

As a dynamic process, DACD is actually a cyclic operation with repeated filling and dumping of the reboiler every 119.7 hours. So the dynamic-accumulative operation policy for continuous distillation (DACD) is feasible and operable.

## OPTIMAL OPERATION

### Important factors for DACD

DACD has several advantages in the purification process of anisole where a small amount of heavy impurities to be removed compared to conventional schemes. On the one hand, it could solve the difficulty of flow rate control when the bottom flow rate is very small in continuous distillation. On the other hand, the demand of still size is also reduced than that in batch distillation. There are two factors that may be very important for the optimal operation of dynamic-accumulative policy for continuous distillation, which are:

1) Initial reboiler holdup;
2) The time to start the second period.

In the following, we will consider the two optimization parameters and the objective is to present guidelines for the purification of anisole in the course of $B^{10}$ enrichment.

### Optimization results

The effect of initial reboiler holdup on product purity is studied by simulation. As Figure 4 and Figure 5 illustrate, the larger the initial reboiler holdup is set, the longer the first period and each cycle last. Therefore, when handling the same amount of total feed, the increasing of the initial reboiler holdup can reduce the number of cycle operations significantly. Equation (10) is used to calculated the recovery ratio of anisole. The yields of anisole are all over 99.99% under different initial reboiler holdup.

$$\eta = \left[1 - \frac{Wt_2\bar{x}}{Fx_F\left(t_1 + t_2 + t_3\right)}\right] \times 100\% \tag{10}$$

However, the investment cost will increase if the still size is too big. Besides, a larger initial reboiler holdup can cause a lower purity of anisole in top at the end of the second period which affects the average concentration of product. In the simulation calculation, the flow rate of the bottom stream in the second period is specified as a constant value, so more reboiler holdup leads to more time consumption in the second period. When time consumption increases to a certain degree, the purity of anisole in top comes down and fails to meet the product requirement. So it exists a limit value for the initial reboiler holdup. As shown in Figure 4, each additional 10 kg of the reboiler holdup brings additional about 126 hours of operation time. When the initial reboiler holdup is 30 kg, the anisole concentration in top is 0.9969 (below 0.997). Therefore, the specified initial reboiler holdup is chosen as 30 kg. Of course we can change the flow rate of the bottom stream in the second period to change time consumption in industrial operation. So we could make a comprehensive consideration on operation convenience, handling amount and actual demands to choose an optimal value of initial reboiler holdup in industrial production.

In order to ensure the distillate reaches its on-specification composition, we must drain away the bottom residue in time when the top concentration dropped to 0.997. However, there is such a tiny difference in the boiling point of the top product at atmospheric pressure when the concentration in top dropped from 0.9975 to 0.997 (about 0.0136°C). In practice, we cannot decide the time when the second period begins according to the temperature variation on the top of rectification column. But we can decide the time according to the bottom temperature as the concentration in bottom varied greatly. Figure 6 shows the relations between the concentration of bottom residue when the second period begins and the recovery ratio of anisole. As can be seen from Figure 6, the concentration of the bottom residue when the second period begins should not be greater than 12.2% to ensure that the yield of anisole is more than 99.9%.

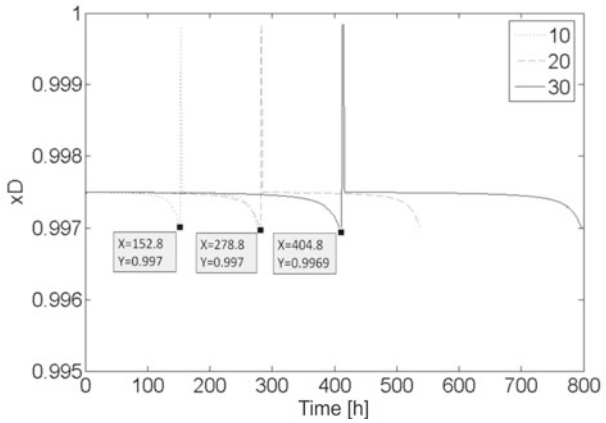


**Figure 4.** Effects of initial reboiler holdup on concentration in top

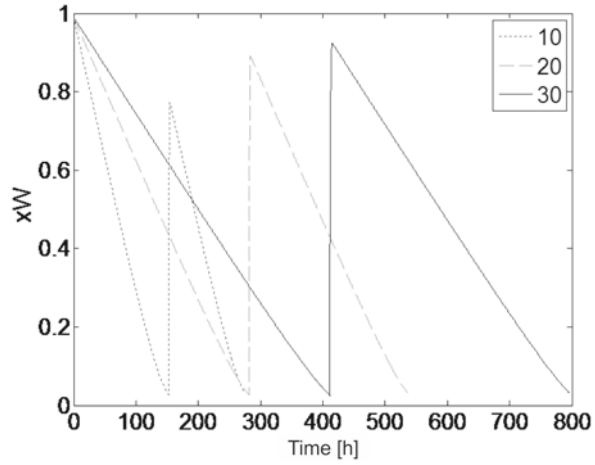


**Figure 5.** Effects of initial reboiler holdup on concentration in bottom

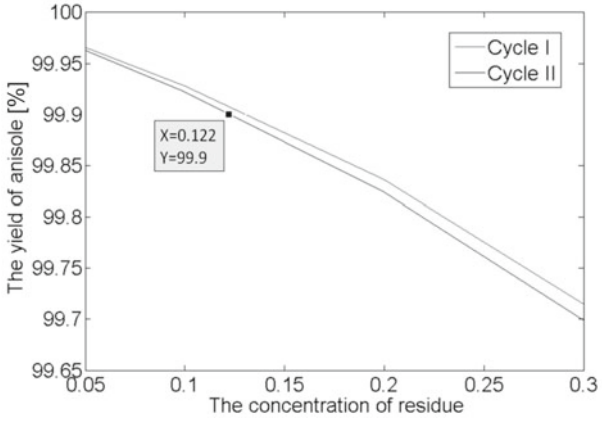


**Figure 6.** Effects of residual concentration on the yield of anisole

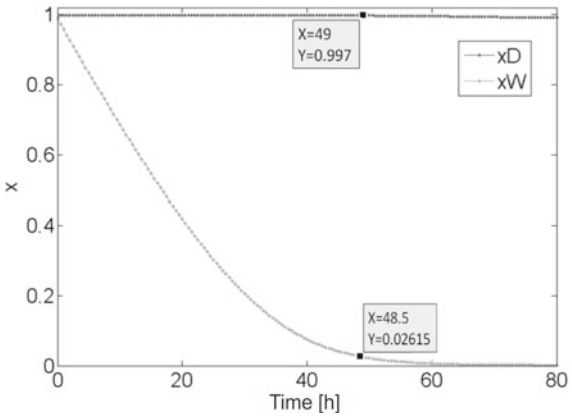


**Figure 7.** Concentration profile in top and bottom with time (MB = 2 kg)

The amount of residue removed from the reboiler in the second period is bound to affect the next cycle. The more the residue removed from the reboiler in the second period, the longer the next cycle lasts. But we should keep the reboiler holdup at a certain level at the end of the second period to make the vapor flow rate from the reboiler remain stable during the whole process, so the specified amount of the residue removed from the reboiler is 28 kg. During the second period, the bottom holdup ($M_B$) decrease from 30 kg to 2 kg. In order to determine the time point when the second period begins, we carried out an additional simulation when $M_B$ is 2 kg. Figure 7 shows concentration profile in top and bottom with time when $M_B$ is 2 kg. As shown in Figure 7, the anisole concentration on the top decreases to 0.997 after 49 hours. If we start the second period (dumping out the heavy impurities) at this moment, the anisole concentration on the top will drop below 0.997 owing to the time consumption for the second period, although it's a short time. Therefore, in order to ensure the product concentration, we start the second period 30 minutes early. That is to say, 30 minutes is set aside for the second period to be in progress and we start the second period at 48.5th hour. The less $M_B$ is, the faster the anisole concentration on the top decreases to 0.997, so if 30 minutes is sufficient when $M_B$ is 2 kg, it's more sufficient when $M_B$ is 30 kg. Therefore the concentration of bottom residue when the second period begins has a minimum value of 2.615% (see Fig. 7). Therefore, the concentration range of the bottom residue when the second period begins is 2.615%~12.2%. Having balanced the concentration in top against the yield of anisole, we start the second period when the concentration of the bottom residue is 10%. Table 2 summarizes the simulation results using these optimized parameters, the calculated recovery ratio of anisole is 99.92% according to equation (10).

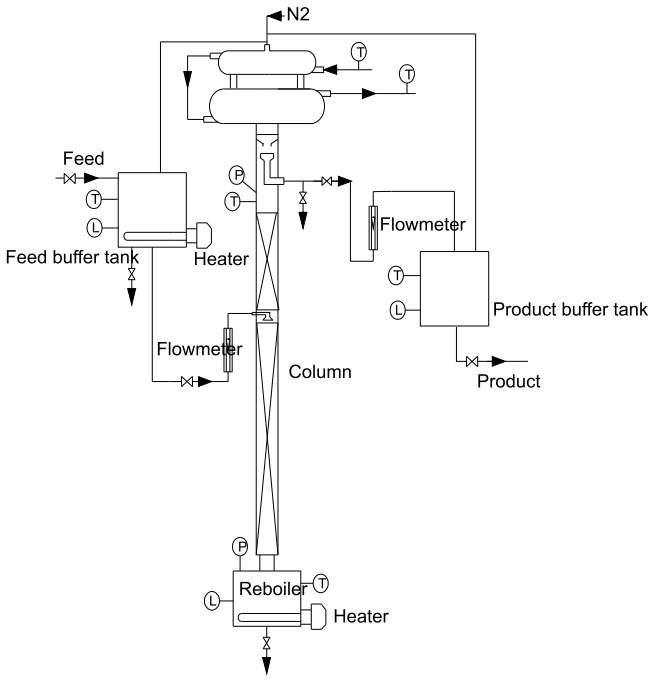


**Figure 8.** Dynamic-accumulative continuous distillation column

## THE EXPERIMENTIAL SECTION

### Experimental apparatus and procedure

The pilot-scale continuous distillation column used in this study is shown schematically in Figure 8. It consists of a 30 liter reboiler, a condenser, and two buffer tanks with a maximum holdup of 30 kg, a packed bed distillation column where the packing height corresponds to 35 theoretical trays and the inner diameter is 80 mm. 6 mm × 6 mm Dixon gauze ring packing is used and the HETP is 0.12 m, the rectifying section has 19 theoretical plates and the stripping section has 16 theoretical plates. Two heat-resistant flow meters accurately measure the flow rate of the feed and the distillate which are controlled by valves. There are temperature measurements on buffer tanks as well as in the reboiler and around the condenser. When the condensate flowed to the product buffer tank, samples are taken off through a manual valve. Similarly, samples of the reboiler and the feed contents can also be collected. The top and bottom pressure of the column are measured by precision pressure meters. The equipment is heated by several coils near the bottom of the reboiler and when the reboiler holdup is 2 kg the coils can be totally immersed in the bottom liquid.

Internal reflux is used in the purification column, in order to estimate actual vapor flow rate from the reboiler, the curve of vapor flow rate from the reboiler and the total pressure drop according to the experiment data is given in Figure 9. The vapor flow rate from the still is measured by the distillate flow meter when the reflux ratio is zero. It shows that the total pressure drop changes very fast when the boilup rate exceeds 22 kg $h^{-1}$. In this experiment, the needed vapor flow rate is 20 kg $h^{-1}$ and the corresponding total pressure drop is 467 Pa.

**Table 2.** Optimization results

| | The First Period [Cycle I] | The Second Period | The Third Period | The First Period [Cycle II] |
|---|---|---|---|---|
| Final Concentration in Top | 0.9974 | 0.9974 | 0.9998 | 0.9974 |
| Final Concentration in Bottom | 0.1006 | 0.09786 | 0.9489 | 0.1001 |
| Operation Time [h] | 372 | 0.5 | 2.8 | 355 |

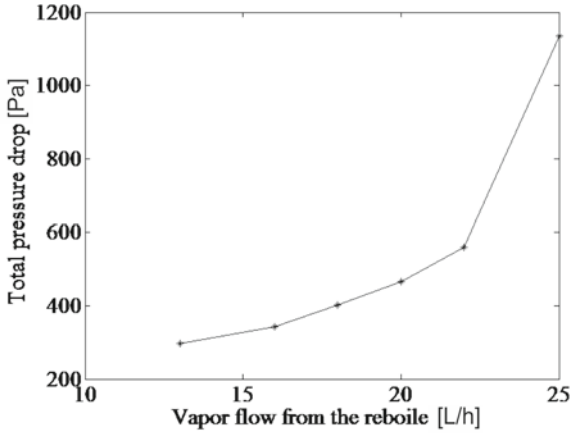


**Figure 9.** The curve of vapor flow from the reboiler and the total pressure drop

At start of the first period, the still pot is filled up with saturated feed (T ≈ 153.8°C) which has been heated in the feed buffer tank. Providing a heat input to the reboiler, the column is started up with total reflux and then the total pressure drop is keep at 467 Pa by adjusting the heater. In this experiment, the end of the total reflux period is taken to be when the temperature variation in the column maintains no more than ±0.1°C over 5 minutes. Once steady state is reached after about 1 hour, the distillate flow rate and the feed flow rate are kept at 10 kg $h^{-1}$ and the bottom flow rate is 0 kg $h^{-1}$. When the concentration in the reboiler decreases to 0.1, the reboiler valve is opened and the bottom residue is being drained out until the bottom holdup decrease to 2 kg. At the same moment the flow rate of the feed and the top product stream as well as reboiler duty remain constant. Then the total reflux operation starts, and the liquid holdup of the bottom is accumulated. A new enrichment cycle begins after the accumulated liquid in the reboiler reaches 30 kg. These three steps are repeated during the purification process of anisole. Record the temperature and concentration in top and bottom in time. The concentration in the experiment is measured by the gas chromatograph.

**Experimental Results and discussions**

In this experiment, we estimate the dynamic changes in the column by observing the temperatures. Figure 10 shows the practical temperatures in top and bottom as a function of time. During the whole process, the overhead temperature remains in the range of 153.9 ± 0.1°C, while the bottom temperature gradually increases with time. After about 367 hours the temperature in bottom rises to 179.5°C and the concentration in bottom decreases to 0.979. The reboiler holdup is partially drained away in 30 minutes and the temperature fluctuation in column is very small during this period of time. Stop drawing out when the bottom holdup decreases to 2 kg. The reboiler filling period lasts for 2.8 hours. During this period, the overhead temperature decreases to 153.8°C and the bottom temperature drops from 179.6°C to 155.4°C. In the second cycle, the temperature in bottom rises to 179.5°C again after 345.7 hours.

During the whole process, the overhead temperature and concentration remain basically invariable. The bottom concentration changing with time is presented in Figure 11. Obviously, experimental data agreed well with simulation results. After testing, the concentration of the anisole in the product buffer tank is greater than 0.9971 and the calculated recovery ratio of anisole is 99.91% according to equation (10).

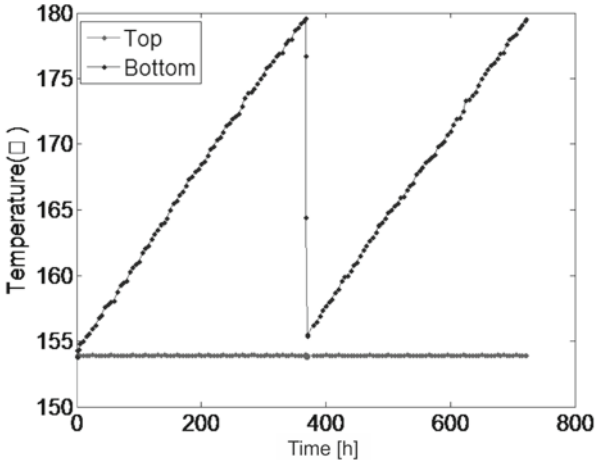


**Figure 10.** Temperature profile in a pilot-scale continuous column using a dynamic-accumulative operation policy (Mixture: anisole (99 wt%), 4-methylanisole (0.2 wt%), 2-methylanisole (0.2 wt%), phenol (0.6 wt%))

Once the column has started up, the operation is only to dump and fill the reboiler about every 345.7 hours. The first circle lasts about 367 hours and each following circle lasts about 345.7 hours. Besides, since 10% is chosen as the concentration of the bottom residue when the second period starts and the corresponding temperature in bottom is 179.5°C according to the experiment, the bottom residue should be drawn out when the temperature in bottom is 179.5°C. Therefore, one attractive feature of DACD is that it is relatively simple and convenient to operate and control. Another advantage of DACD is that the size of the still can be smaller than that in batch distillation because of charging the feed continuously.

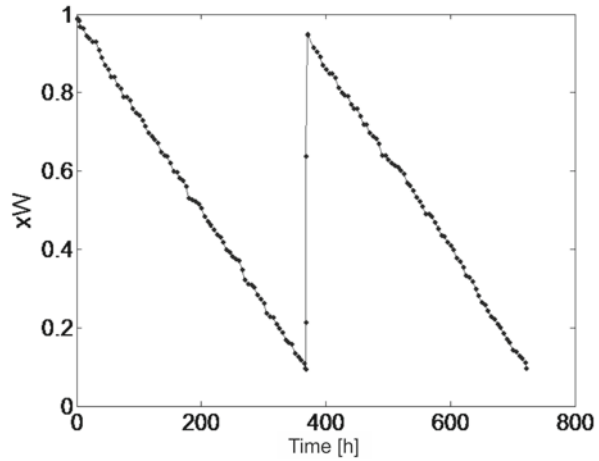


**Figure 11.** Concentration profile in a pilot-scale continuous column using a dynamic-accumulative operation policy (Mixture: anisole (99 wt%), 4-methylanisole (0.2 wt%), 2-methylanisole (0.2 wt%), phenol (0.6 wt%))

## CONCLUSIONS

DACD was found to be a favorable operation mode in the purification process of anisole where a large amount of light product to be recovered. It not only solved the difficulty of flow rate control when the bottom flow rate was very small in continuous distillation, but also made the still size smaller than that in batch distillation. In addition, the residue which contained high levels of heavy impurities was drained away after a long period of time. The yield of anisole was increased to 99.91%.

The feasibility and practicality of DACD were proved by the experiment. DACD promises to be very useful for the separation with a large amount of light component i to be recovered in pharmaceutical and specialty chemical industries to achieve the most efficient, environmentally friendly and profitable separation process.

## NOMENCLATURE

D – distillate flow rate, [kg $h^{-1}$]
F – feed flow rate, [kg $h^{-1}$]
L – reflux flow rate, [kg $h^{-1}$]
L′/V′ – liquid/vapor flow rate in the stripping section [kg $h^{-1}$]
M – tray holdup [kg]
$M_0$ – condenser holdup [kg]
$M_B$ – initial reboiler holdup [kg]
$M_B''$ – reboiler instantaneous holdup in the third period [kg]
$N_f$ – the position of the feeding theoretical plate
$N_t$ – number of theoretical plates in column section
R – reflux ratio
V – vapor flow rate in the rectifying section [kg $h^{-1}$]
W – flow rate of the bottom stream in the second period [kg $h^{-1}$]
$\bar{x}$ – the average concentration of the residue removed from the still
$x_B$ – liquid composition in the still
$x_j$ – liquid composition on theoretical plate *j*
$x_{j,i}$ – liquid composition of component *i* on theoretical plate *j*
$y_j$ – vapor composition on theoretical plate *j*
$y_{j,i}$ – vapor composition of component *i* on theoretical plate *j*
$z_j$ – feed composition

**Greek symbols:**

α – relative volatility
ω – reboiler holdup at the end of the second period [kg]

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