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# Student-led Inquiry Learning Ability Training - Taking Distillation Experiment as An Example


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## Abstract

Inquiry-based study can enhance long term retention and improve application and synthesis of knowledge. In this research, we provide a student-driven, inquiry-based teaching model that trains undergraduate as researcher, who can raise questions, design and perform hypothesis-driven experiments, analyze data, and discuss results. Before students design their research projects, they should know well and answer a series of questions related to experiment principle, condition and error control, data detection, and processing, which guide them to familiarize with project principles, operations, and data processing. Continuous distillation is an ideal choice for the training in an undergraduate lab due to its synthesizing the fluid flow, heat transfer, and mass transfer. Students actively participate in the project, because their doubts can be removed through designing and implementing experiments, such as: How to evaluate the separation capacity of the distillation operation? How to implement the experiment and get the component concentration in a simple way? How to evaluate the separation capacity of the packing column? Students use their acquired research skills to design, execute, and analyze experiments independently. While, the instructors only make some adjustments to ensure that the experimenter completes the design goal within the limited experimental time. Results show that the distillation experiment builds an open, inquiry-based platform for students, which significantly enhances the ability to apply and integrate knowledge. The scientific research ability and teamwork awareness have also been nurtured.



**Keywords:** Inquiry mode; Student-driven; Chemical engineering; Distillation experiment.




## 1. Introduction

Student interests, attitudes and studying method have a significant impact on their learning outcomes. Inquiry-based mode can enhance long term retention, and improve synthesis application of knowledge. It can also provide valuable opportunities for students to improve their understanding of both science content and scientific practices (Edelson *et al.*, 1999; Goldey *et al.*, 2012; Liu, 2018). In the training of undergraduate as scientists, the inquiry-based teaching mode of pose questions, design project and implement research to answer questions, and subsequently draw conclusions from data is an effective approach (Lemons, 2016; Triyanto, 2018), especially for experimental courses. This is a challenge to the traditional teaching mode of simple introduction or verification according to the established experimental protocol. Furthermore, it is a thoroughly overthrow for the "standardization" teaching materials, "commoditized" equipment and "cramming" teaching mode, which is difficult to provide students with space and time to think. Therefore, it is very important to introduce the inquiry-based teaching mode into experimental course teaching.

In this research, we describe a student-driven experiment project in undergraduate lab teaching of distillation. Distillation is a typical mass transfer unit operation between the ascending vapor phase and the reflux liquid phase based on the difference in volatility of liquid components. A liquid mixture of component A and component B can be separated into individual components or into groups of components through repeated heat transfer and mass transfer on the surface of the distillation column or packing (Górak and Sorensen, 2014). For students majored in chemistry or other related profession, distillation is one of the obligatory content in both the "Chemical Engineering" theoretical teaching and experimental teaching (McCabe *et al.*, 1993; Wuhan University *et al.*, 2005; Wuhan University, 2016). In particularly, the separation capacity is related to various factors such as physical factors, equipment factors (plate or packing type) and operating conditions (distillation still pressure and the corresponding


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vapor volocity, liquid volocity; reflux ratio, feed temperature, etc). As for the equipment, it contains the measurement and control instruments of temperaturer, piezometer, flowmeter, liquidometer; and the quantitative analysis of the bottom product and overhead product is also need. Furthermore, distillation integrates the contents of momentum transfer, heat transfer and mass transfer. Therefore, distillation experiment is suitable as a design project to examine students' synthesis capabilities of design, implementation, condition control, data processing and results analysis. The project uses a positive and student-driven mode, and sets two overall goals: 1) Training students as scientists to design, execute and analyze research projects. 2) Encourage students to gain a deep understanding of distillation unit operation by applying their knowledge to interpret the meaning of experimental results.

## 2. Course Background

The student-driven, inquiry-based instruction mode is carried out in Chemical Engineering Experiment course for about 300 undergraduates, and distillation is one of the obligatory content. Within four weeks, all students, regardless of their major, must master distillation concept and the related equipment through self-study. Instructors supply related learning materials (Ma and Pang, 2017; McCabe *et al.*, 1993; Wuhan University L. U. a. F. U., 2005; Wuhan University, 2016), and give lectures of 45 minutes and 2 times per week. In this period of time, students are required to learn the distillation principle, equipment, calculation of the number of ideal plates and height equivalent to a theoretical plate (HETP). Then, the experimental exercise lasts for a total of 6 weeks, which is 6 times a week with a 4-hour time block. Each time there are four groups, each group of 2 students carry out their experimental project. The instructor intentionally selects the groups to ensure that a combination of different major backgrounds is represented in each group.

## 3. Implementation

### 3.1. Preparation for Lab Teaching

To carry out the inquiry-based mode, instructors prepared four packing-column instruments supplied by the equipment manufacturer. These equipment was filled with the same filler (as shown in Figure 1), and it can be used to investigate the influence of different operational factors on separation capacity. In order to increase designability and diversity, instructors made the following preparation and improvement.

#### 3.1.1. Bicomponent Distillation System

Based on the concept of less pollution and energy-saving, the ethanol and n-propanol solutions were prepared as separated mixture, and the gas-liquid equilibrium data were offered as Table 1 (Zhang *et al.*, 2005). Abbe refractometer was employed to provide quantitative analysis of distillate and residue.

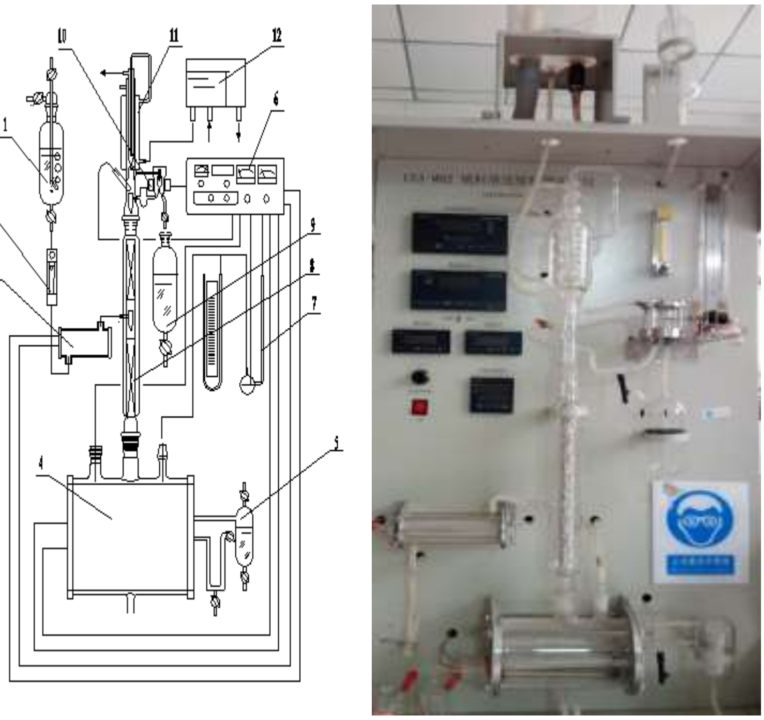
#### 3.1.2. Experimental Apparatus

In view of the effect of packing on separation capacity, instructors provided four different packings of stainless steel calendered $\theta$-ring (Figure 2 (a)), ceramic raschig (Figure 2 (b)), glass-spring packing (Figure 2 (c)) and wire mesh $\theta$-ring (Figure 2 (d)) (Schultes, 2014). Experimenters can select different distillation column to investigate the influence of packing specific surface area on the results.

#### 3.1.3. Operational Factors

The distillation apparatus can provide the measurement and control of the reflux ratio, the temperature and flow rate of the feed solution, distillation still pressure and the corresponding vapor velocity and liquid velocity. The influence on separation capacity can be investigated according to the experimental needs.

**Figure-1.** The schematic diagram of distillation apparatus

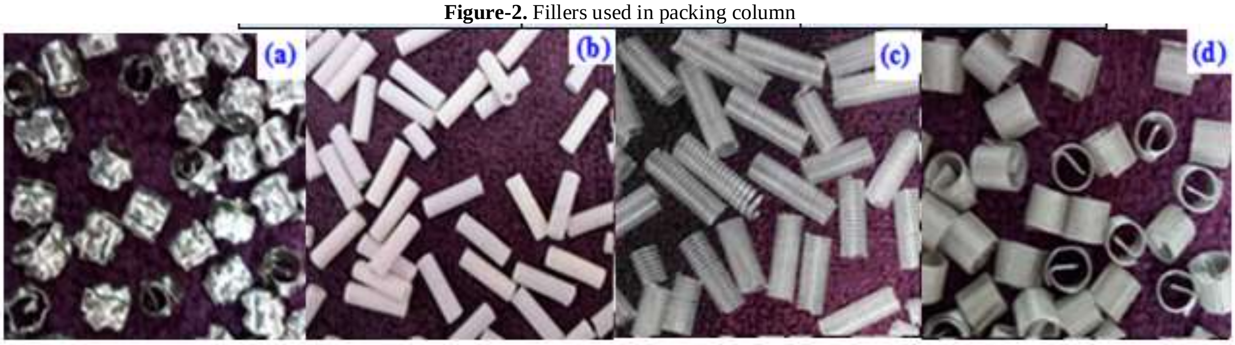


1. head tank of feed solution; 2. flowmeter; 3. preheater of feed solution; 4. distillation still; 5. residue collector; 6. controller; 7. U type differential gage; 8. packing column; 9. distillate collector; 10. reflux ratio regulator; 11. condenser; 12. head tank of cooling water

**Table-1.** The gas-liquid equilibrium data of ethanol and n-propanol

| Temperature °C | $x_A$ (Ethanol molar fraction in liquid) | $y_A$ (Ethanol molar fraction in vapor) |
|---|---|---|
| 97.16 | 0 | 0 |
| 93.85 | 0.126 | 0.240 |
| 92.66 | 0.188 | 0.318 |
| 91.6 | 0.210 | 0.339 |
| 88.32 | 0.358 | 0.550 |
| 86.25 | 0.461 | 0.650 |
| 84.98 | 0.546 | 0.711 |
| 84.13 | 0.600 | 0.760 |
| 83.06 | 0.663 | 0.799 |
| 80.59 | 0.844 | 0.914 |
| 78.38 | 1.0 | 1.0 |

**Figure-2.** Fillers used in packing column




(a) stainless steel calendered $\theta$-ring ; (b) ceramic raschig ; (c) glass-spring packing; (d) wire mesh $\theta$-ring

## 3.2. Self-study Check

Before the operation exercise, students are demanded to answer the related questions shown in Table 2 in a self-testing manner, and the score is a test of their self-study result. The purpose is to make the experimenter aware of: what projects can I design? How to reveal the influence of operational condition on separating capacity? How to evaluate the separation capacity of different packing, and as for number of theoretical plate and HETP, which is the better evaluation index? If they can get a score more than 80, students can design experiment projects independently and then implement it.

**Table-2.** Self-test

| Number | Question |
|---|---|
| 1 | What are the main factors affecting the separation capacity of distillation? ( )<br>A. Physical properties of the components; B. Distillation equipment;<br>C. Operational conditions. |
| 2 | How to control the vapor velocity and liquid velocity in the packing column? ( )<br>A. Control the heating power of distillation still; B. Control the reflux ratio; C. Control the flow of cooling water; D. Control the pressure in distillation still. |
| 3 | For the same distillation apparatus, the evaluation indexes for separation capacity are ( ).<br>A. The number of theoretical plate ($N_T$); B. *HETP*; C. Purity of the distillate. |
| 4 | When evaluating the separation capacity of packing columns with different heights, the evaluation index is ( ).<br>A. The number of theoretical plate $N_T$; B. *HETP*; C. Purity of the distillate. |
| 5 | When evaluating the separation capacity of packing columns, how to ensure that the experimental conditions are parallel ( )?<br>A. Control the same operational conditions such as vapor velocity, liquid velocity and reflux ratio;<br>B. the feed solution with the same components, concentration and temperature;<br>C. The consistent filling method of packing. |
| 6 | How to judge that the separation process has reached equilibrium? ( )<br>A. Temperature, pressure and other parameters remain unchanged;<br>B. The distillate content (refractive index) remains unchanged;<br>C. The separation keeps enough time. |
| 7 | What is the reflux ratio, and how to set its value? |
| 8 | How to measure and control the flow rate and temperature of feed solution? |
| 9 | How to measure and control the distillation column temperature? |
| 10 | Does temperature have any effect on the determination of refractive index? How to eliminate the effect? |
| 11 | In quantitative determination with Abbe refractometer, how to reduce volatilization loss and composition change during product cooling? |
| 12 | What is the necessity of liquid reflux in a continuous distillation? |
| 13 | What is the relationship between the distillation still pressure and the vapor velocity in packing column? How to measure and control it? |
| 14 | To get $N_T$ or *HETP*, what parameters need to be detected? How to process this data? |
| 15 | How to ensure the experiment safety? |

## 3.3. Experimental Project Design

Based on the understanding of the distillation principle and apparatus, students designed a variety of experimental projects, and they were mainly divided into two types of independent project and synthesis project. In independent project, the operators could carry out the experiment with any one of the apparatus to investigate the separation capacity of packing columns at different operational conditions, such as different vapor velocities and corresponding liquid velocities, different reflux ratio, different feed solution temperature. In independent experiment, students could only investigate the influence of single factor on separation capacity, and get the confirmatory results and conclusions. In synthesis projects, the operators could carry out experiments with any two, three or four of the apparatus to investigate the separation capacity of different packing columns at different operation conditions, and get synthesis results and conclusions.

### 3.3.1. Independent Project Design

In the investigation of operating conditions on separation capacity of packing column, the designed projects are listed as follows.

Project 1: Influence of vapor velocity and corresponding liquid velocity on separation capacity.

Project 2: Influence of reflux ratio on separation capacity.

Project 3: Influence of feed solution temperature on separation capacity.

### 3.3.2. Synthesis Project Design

With any two, three or four of the apparatus, investigate the separation capacity of different packing columns at different operational conditions. Implementation of these projects can cultivate the teamwork ability among students. The designed projects are classified and listed as follows.

Project 1: Performance evaluation of stainless steel calendered $\theta$-ring packing columns and ceramic raschig packing columns

Project 2: Performance evaluation of stainless steel calendered $\theta$-ring packing columns and glass-spring packing packing columns

Project 3: Performance evaluation of stainless steel calendered $\theta$-ring packing columns and wire mesh $\theta$-ring packing columns

Project 4: Performance evaluation of ceramic raschig packing columns and glass-spring packing packing columns

Project 5: Performance evaluation of ceramic raschig packing columns and wire mesh $\theta$-ring packing columns

Project 6: Performance evaluation of glass-spring packing packing columns and wire mesh $\theta$-ring packing columns

Project 7: Performance evaluation of stainless steel calendered $\theta$-ring packing columns, ceramic raschig packing columns and glass-spring packing packing columns

Project 8: Performance evaluation of stainless steel calendered $\theta$-ring packing columns, ceramic raschig packing columns and wire mesh $\theta$-ring packing columns

Project 9: Performance evaluation of stainless steel calendered $\theta$-ring packing columns, glass-spring packing packing columns and wire mesh $\theta$-ring packing columns

Project 10: Performance evaluation of ceramic raschig packing columns, glass-spring packing packing columns and wire mesh $\theta$-ring packing columns

Project 11: Performance evaluation of packing columns packing with different filler of stainless steel calendered $\theta$-ring, ceramic raschig, glass-spring packing and wire mesh $\theta$-ring

## 3.4. Experimental Project Execution and Results

Based on the designed project and the provided apparatus, students independently carried out experiments, and instructors provided necessary guidance. From the many design projects, three typical cases were selected and described in detail.

**Project 1: Influence of vapor velocities and liquid velocities on separation capacity.**

In the condition of total reflux, students carried out experiment under different distillation still pressure and corresponding vapor velocities, liquid velocities.

Packing: wire mesh $\theta$-ring; Height: 0.316 m; Flooding pressure $P_{max}$: 8.7cm$H_2O$; Components: mixture of ethanol and n-propanol with a volume ratio of 1:3; Refractive index: $n_{d,ethanol}^{15} = 1.3645; n_{d,propanol}^{15} = 1.3882$

The recorded raw data were listed in Table 3. Based on the gas-liquid equilibrium relation shown in Table 1, students calculated the number of theoretical plates ($N_T$) and the height equivalent to a theoretical plate (*HETP*) according to equation (1)~(3), and got the results as shown in Table 4. They further gave the conclusions of: (1) Wire mesh $\theta$-ring packing column indicated the biggest $N_T$ and smallest *HETP* at 3.5 cm$H_2O$, and presented the highest separation capacity. (2) For the separation of ethanol-n-propanol mixture with wire mesh $\theta$-ring column, it could be carried out at distillation still pressure of 3.5 cm$H_2O$ to get the highest purity of overhead product and bottom product.

$$N_{\mathrm{T}} = \frac{\ln\left[\left(\frac{x_d}{1-x_d}\right)\left(\frac{1-x_w}{x_w}\right)\right]}{\ln\alpha} - 1 \tag{1}$$

$N_T$ : number of ideal plate;

$x_d$ : mole fraction of the more volatile component in overhead product;

$x_w$ : mole fraction of the more volatile component in bottom product;

$\alpha$ : relative volatility.

$$n_{d,mixture} = n_{d,ethanol} \cdot x_{ethanol} + n_{d,propanol} \cdot (1 - x_{ethanol}) \tag{2}$$

$n_{\mathrm{d}}$ : refractive index.

$$HETP = \frac{h}{N_{\mathrm{T}}} \tag{3}$$

*HETP*: the height equivalent to a theoretical plate; *h*: packing height.

**Table-3.** Influence of vapor velocities and liquid velocities on separation capacity

| Pressurein distillation still $P$/cm$H_2O$ | Temperature of vapor/°C | Temperature of residue/°C | Refractive index of distillate | Refractive index of residue |
|---|---|---|---|---|
| 1.7 | 62.3 | 94.8 | 1.3700 | 1.3831 |
| 3.5 | 65.5 | 95.9 | 1.3690 | 1.3852 |
| 5.2 | 63.3 | 95.3 | 1.3713 | 1.3849 |

**Table-4.** Results of $N_T$ and *HETP*

| Pressure in distillation still $P$/cm$H_2O$ | $x_d$ | $x_w$ | $N_T$ | *HETP*/m |
|---|---|---|---|---|
| 1.7 | 0.767 | 0.216 | 2.2 | 0.144 |
| 3.5 | 0.810 | 0.0286 | 5.3 | 0.060 |
| 5.2 | 0.712 | 0.137 | 2.5 | 0.126 |

**Project 2: The influence of reflux ratio on separation capacity**

In this project, students designed a series of experiments to investigate influence of reflux ratio on separation capacity in the condition of bubble point feed.

Packing: stainless steel calendered $\theta$-ring; Height: 0.330 m; Flooding pressure $P_{max}$: 15.0 cm$H_2O$; Components: mixture of ethanol and n-propanol with an initial volume ratio of 1:1; Feed temperature: bubble point of 91.2 °C;

Feed rate 10 mL/min; Pressure in distillation still: 7.0 cm$H_2O$; Refractive index: $n_{d,ethanol}^{18} = 1.3630; n_{d,propanol}^{18} = 1.3866$

The experimenters recorded the raw dada (as listed in Table 5), calculated $N_T$, *HETP* and utilization coefficient $k$ according to equation (4). The results were shown in Table 6 and Figure 3. They further gave the conclusions of: (1) For the packing column of a certain height, the corresponding $N_T$ and $k$ increased with the increase of the reflux ratio, and the *HETP* gradually decreased, which indicated that the separation ability for the ethanol-n-propanol mixture was gradually enhanced. (2) Product purity could be improved by increasing reflux ratio.

$$k = \frac{HETP_{\infty}}{HETP} = \frac{N_T}{N_{T,\infty}} \qquad (4)$$

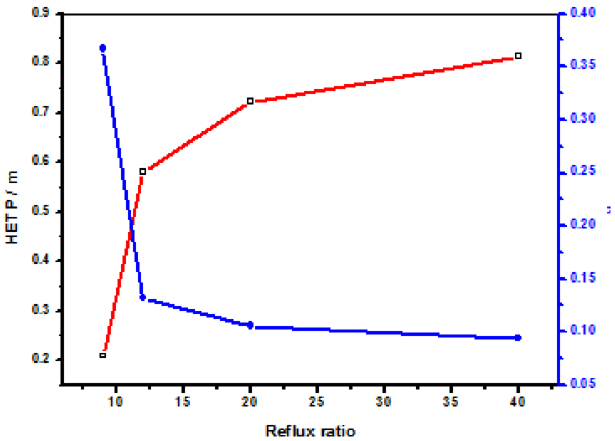
**Table-5.** Data under different reflux ratio

| Reflux ratio | Refractive index of distillate | Refractive index of residue |
|---|---|---|
| 9 | 1.3695 | 1.3768 |
| 12 | 1.3660 | 1.3779 |
| 20 | 1.3650 | 1.3782 |
| 40 | 1.3647 | 1.3787 |
| ∞ | 1.3640 | 1.3790 |

**Table-6.** Results under different reflux ratio

| Reflux ratio | $N_T$ | HETP/m | k |
|---|---|---|---|
| 9 | 0.9 | 0.367 | 0.209 |
| 12 | 2.5 | 0.132 | 0.581 |
| 20 | 3.1 | 0.106 | 0.721 |
| 40 | 3.5 | 0.094 | 0.814 |
| ∞ | 4.3 | 0.0767 | 1 |

**Figure-3.** The influence of reflux ratio on *HETP* and *k*



**Project-3. The influence of the packing on separation capacity**

In this project, students designed experiment to investigate influence of four different packing columns of stainless steel calendered $\theta$-ring, ceramic raschig, glass-spring packing and wire mesh $\theta$-ring on separation capacity in the condition of total reflux operation. They detected the size of packing, fill height, porosity and flooding pressure as listed in Table 7. Other parameters were recorded as:

Components: mixture of ethanol and n-propanol with an initial volume ratio of 1:3; $n_{d,ethanol}^{18} = 1.3630; n_{d,propanol}^{18} = 1.3866$

To ensure consistency of the operational conditions, the evaluation was carried out in the condition of total reflux. The distillation still pressure and its corresponding vapor velocity and liquid velocity were controlled based the minim $P_{max}$ of 10.2 $cmH_2O$ of the four packing columns. 3 $cmH_2O$, 5 $cmH_2O$ and 7 $cmH_2O$ were selected as control condition, and the raw data were recorded as Table 8. Since the four tested packing columns had different height, students selected *HETP* as the evaluation standard and the calculated results were also shown in Table 8. Based on the results, they gave the following conclusions:

(1) Distillation still pressure and the corresponding vapor velocity and liquid velocity showed different influence on different packing, the *p* value corresponding to the best separation capacity was 5.0 $cmH_2O$ for stainless steel calendered $\theta$-ring. As for the other three packings, separation capacity increased with the *p* value increased.

(2) At the same pressure, the separation ability of different packing columns was also different. The stainless steel calendered $\theta$-ring, wire mesh $\theta$-ring, glass-spring packing and ceramic raschig indicated the gradually increasing *HETP* value, and then showed the gradually decreasing separation capacity for ethanol-n-propanol solution with an initial volume ratio of 1:3.

(3) The separation ability was related to both the distillation still pressure (effect the vapor velocity and liquid velocity), and the specific surface area of packing. When the vapor velocity, liquid velocity and specific surface area of packing were all suitable, the greater the effective contact surface area, the higher the separation ability.

(4) For the ethanol-n-propanol mixture with an initial volume ratio of 1:3, the best separation conditions were: stainless steel calendered $\theta$-ring packing column distillated at the distillation still pressure of 5 $cmH_2O$.

**Table-7.** Structure parameters and flooding pressure of the used packings

| **Packing** | **Size (diameter×height) /mm$^2$** | **Filled height/ cm** | **Porosity /%** | **Flooding pressure $P_{max}$ /$cmH_2O$** |
|---|---|---|---|---|
| Ceramic raschig | 3×10 | 31.0 | 34.1 | 10.2 |
| Stainless steel calendered $\theta$-ring | 3×3 | 33.0 | 72.3 | 15.4 |
| Wire mesh $\theta$-ring | 4×4 | 34.0 | 86.5 | 12.2 |
| Glass-spring packing | 4×13 | 38.0 | 78.0 | 11.0 |

**Table-8.** Separation data and results of the four packings

| Packing | *P* /$cmH_2O$ | Refractive index of distillate | Refractive index of residue | *HETP* /m |
|---|---|---|---|---|
| Ceramic raschig | 3.0 | 1.3688 | 1.3811 | 0.155 |
| | 5.0 | 1.3660 | 1.3789 | 0.132 |
| | 7.0 | 1.3656 | 1.3789 | 0.119 |
| Stainless steel calendered $\theta$-ring | 3.0 | 1.3636 | 1.3794 | 0.0635 |
| | 5.0 | 1.3635 | 1.3789 | 0.0516 |
| | 7.0 | 1.3640 | 1.3790 | 0.0767 |
| Wire mesh $\theta$-ring | 3.0 | 1.3665 | 1.3812 | 0.117 |
| | 5.0 | 1.3664 | 1.3815 | 0.113 |
| | 7.0 | 1.3650 | 1.3813 | 0.919 |
| Glass-spring packing | 3.0 | 1.3668 | 1.3811 | 0.141 |
| | 5.0 | 1.3666 | 1.3813 | 0.131 |
| | 7.0 | 1.3655 | 1.3812 | 0.112 |

## 4. Conclusions

Through inquiry-based instruction mode, students can master the principle and condition optimization for distillation separation: Firstly, establishes qualitative and quantitative analysis methods for components. Secondly, obtains relevant data on the relative volatility and temperature. Then, optimizes the separation conditions (such as packing type, distillation pressure, vapor velocity, liquid velocity, etc.) through parallel experiments. Finally, determines the proper packing and operating conditions to get the product with the highest purity.

The mentioned distillation experiment builds an open, inquiry-based platform for students. Taking students as the center and cultivating their ability to master professional knowledge and practice ability is the teaching goal. On this experimental platform, the students can use the knowledge they have learned to design and implement experiment and analyze the results, which significantly enhances the ability to apply and integrate knowledge. The scientific research ability and teamwork awareness have also been nurtured.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