

## Continuous Distillation Experiment with Rotating Packed Bed\*

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**Abstract** The performance of a rotating packed bed (RPB) with three kinds of packings was investigated using alcohol/water solution under continuous distillation conditions at atmospheric pressure. The effects of average high gravity factor ( $\beta$ ), reflux ratio ( $R$ ), and feedstock flux ( $F$ ) on mass-transfer in distillation were examined separately. Experimental results indicated that the total number of theoretical units (NTU) of RPB increased with  $\beta$ ,  $R$ , and  $F$ . Of the three kinds of packings, the wave thread packing of stainless steel (Packing III) had the best mass transfer efficiency with the height equivalent of a theoretical plate (HETP) of approximately 7.35 mm–23.58 mm, whereas the corrugated disk packing of stainless steel (Packing I) had the worst one with the HETP of about 13.4 mm–48.07 mm. Correlations were developed to describe the mass transfer efficiency for packings. Comparing experimental data with the data calculated by correlation, the average deviate obtained for each packing was 0.72%, 1.98%, and 2.7%, respectively, implying that the accuracy of correlations developed was reasonable.

**Keywords** rotating packed bed, continuous distillation, packing, correlation

### 1 INTRODUCTION

Large amounts of organic solvents are used to extract or purify the end products in pilot plants in finery chemicals industry, biological chemicals industry, and food and pharmaceutical industry. These solvents are generally recycled by distillation process. Although large scale distillation units are more economical with less heat loss in operation, it is not economical for small or meso-scale factories to build large distillation unit, because the volume of waste organic solvents is small and most of them are batch or semi-continuous effluent. Still, the specifications of the recycled solvents by simple distillation process are not satisfactory. According to documentation and theoretical analysis, the separation of the above solvents can be conducted with rotating packed bed (RPB) under high gravity field, which is small in size, efficient, easily-operated and flexible.

As a novel mass and heat transfer device, the applications of rotating packed bed have been widely studied by many researchers [1–3] in absorption [4, 5], stripping [6, 7], extracting [8, 9], desulfurizing [10], removing dust [11], making nano-material [12, 13], and so on. Burns *et al.* [14], Matin *et al.* [15], Chen *et al.* [16], Lin *et al.* [17], Li *et al.* [18–20], and Trevour *et al.* [21] have conducted experiments with RPB, using different materials (such as methanol/water mixture, alcohol/water mixture, or cyclohexane/*n*-heptane mixture) at different pressures (mainly at atmospheric pressure) under total reflux condition. Their findings showed that the height equivalent of a theoretical tray of RPB with different packings ranged from 10 mm to 90 mm. However, nothing of a quantitative nature has been published on the use of RPB at some reflux rate in continuous operation. The main objective of the present work is to provide operating information on a scale RPB unit running continuously to separate alcohol/water mixture. Mass transfer efficiency of RPB

under different operating conditions, such as average high gravity factor, reflux ratio, and feedstock flux, with three kinds of packings are investigated, and the semiempirical correlations developed should serve as a framework for further application studies of the RPB.

### 2 EXPERIMENTAL

#### 2.1 Flow diagram

Figure 1 shows the flow diagram of the experimental system. The facility was provided by Research Center of Shanxi Province for High Gravity Chemical Engineering and Technology. As shown, the two-RPB system is configured for continuous operation at atmospheric pressure with the upper one (RPB2) serving as rectifying section and the lower one (RPB1) as stripping section. During operation, a predetermined amount of feedstock is charged initially into the container. The feedstock is pumped into RPB1 through a flowmeter testing its feed flow rate. Liquid flows into

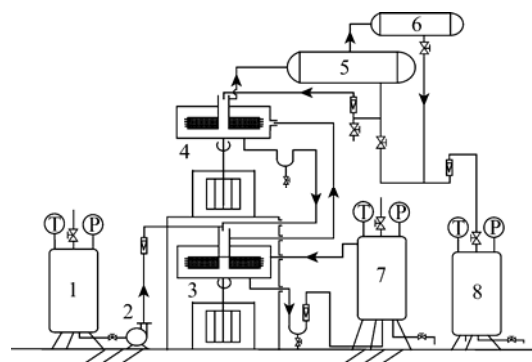


**Figure 1** Schematic diagram of the experimental setup  
1—container; 2—pump; 3—RPB1; 4—RPB2; 5,6—condenser; 7—reboiler; 8—recipient

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a distributor comprising two vertical tubes with holes drilled in them. Liquid leaves the distributor at a relatively high velocity and then flows into the inner edge of the rotor. The velocity of the liquid exhausted from the distributor must be sufficiently high to pass the space between the distributor and the inner edge of the rotor. Otherwise, liquid entrainment might occur in the discharged vapor and the liquid maldistribution would subsequently appear within the rotor. Liquid moves outward because of centrifugal force in rotor, and then, leaves the rotor at the outer edge. Liquid is expelled from the bottom of the RPB1 and flows into the re-boiler, in which some liquid is discharged as waste and the other heated into vapor back to RPB1.

In RPB1, vapor travels inward from the outer edge of the rotor by the pressure driving force, contacting with liquid counter-currently to transfer heat and mass, and leaves the rotor at the inner edge, then is discharged through the top of the RPB1 into RPB2. Vapor from RPB2 is condensed into a condensate against cooling water in overhead condenser. The condensate is divided into two parts, with one part returned to RPB2 as reflux and the other discharged as overhead product.

## 2.2 Physical properties of the rotor and packings

The rotor and packings in it are major sections for RPB and play an important role in distillation process. Physical properties for the rotor and packings used in experiment are shown in Table 1 and Table 2.

Table 1 Physical properties for the rotor

| O.D./mm | I.D./mm | Height in axis/mm | Porosity | Material        |
|---------|---------|-------------------|----------|-----------------|
| 110     | 60      | 63                | 0.68     | steel-stainless |

## 2.3 Test conditions

The alcohol/water binary was used as the test mixture with the concentration of alcohol of approxi-

mately 10%–15% (mass) for feedstock. Feed flow rate was about 0–30 L·h<sup>-1</sup>. The rotor could be rotated from 0 to 1830 r·min<sup>-1</sup>.

In operation, temperatures of gas and liquid were measured by thermometers. Concentration of mixture was measured by a gas chromatography of SQ206, and the flow flux of liquid was monitored by a flowmeter. Rotating speed of rotor was modulated by a governor motor (Type: Y80M1-265, Power: 0.75kW).

## 3 RESULTS AND DISCUSSION

By way of observation, the RPB system operated smoothly and trouble-free during the experimental period. No instabilities resulting from rotation were observed.

### 3.1 Mass transfer performance of RPB

Separation efficiency may be presented in terms of the number of transfer units (NTU) achieved. Using Huffman formula [22], NTU was calculated from the following Eqs. (1) and (2), where  $A = R(1 - \alpha)$ ,  $B = R(\alpha - 1) - x_D(\alpha - 1) + \alpha$ ,  $A' = R'(1 - \alpha)$ ,  $B' = R'(\alpha - 1) - x_D(\alpha - 1) + \alpha$ ,  $R' = [(x_D - x_W) + R(x_F + x_W)] / (x_D - x_F)$ . In this expression,  $A$ ,  $B$ ,  $A'$ , and  $B'$  are parameters,  $\alpha$  is a relative volatility,  $x_D$ ,  $x_W$ , and  $x_F$  represent the concentration of upper tower liquid, bottom liquid, and feedstock, respectively.

The functional trend of NTU with the operating parameters, such as average high gravity factor ( $\beta$ ), reflux rate ( $R$ ), and feedstock flux ( $F$ ), is the main item of concern.

#### 3.1.1 The effect of $\beta$ on NTU for RPB

As a dimensionless factor, high gravity factor ( $n$ ) is defined as a ratio of acceleration of liquid in packing to gravity acceleration:

$$n = \frac{\omega^2 r}{g} \quad (3)$$

where,  $\omega$  denotes rotating speed (r·min<sup>-1</sup>),  $r$  is radius

$$\text{NTU(rectifying section)} = \frac{2R + B}{2\sqrt{B^2 + 4Ax_D}} \ln \frac{(2Ax_0 + B - \sqrt{B^2 + 4Ax_D})(2Ax_F + B + \sqrt{B^2 + 4Ax_D})}{(2Ax_F + B - \sqrt{B^2 + 4Ax_D})(2Ax_D + B + \sqrt{B^2 + 4Ax_D})} + \frac{1}{2} \ln \frac{Ax_F^2 + Bx_F - x_D}{Ax_D^2 + Bx_D - x_D} \quad (1)$$

$$\text{NTU(stripping section)} = \frac{2R' + B'}{2\sqrt{B'^2 - 4A'x_W}} \ln \frac{(2A'x_F + B' - \sqrt{B'^2 + 4A'x_W})(2A'x_W + B' + \sqrt{B'^2 + 4A'x_W})}{(2A'x_W + B' - \sqrt{B'^2 + 4A'x_W})(2A'x_F + B' + \sqrt{B'^2 + 4A'x_W})} + \frac{1}{2} \ln \frac{A'x_W^2 + B'x_W - x_W}{A'x_F^2 + B'x_F - x_W} \quad (2)$$

Table 2 Physical properties for packings

| Packing                             | Diameter/mm | Packing density/kg·m <sup>-3</sup> | Specific surface area/ m <sup>2</sup> ·m <sup>-3</sup> | Porosity | Material        |
|-------------------------------------|-------------|------------------------------------|--------------------------------------------------------|----------|-----------------|
| Packing I (corrugated disk packing) |             | 950                                | 400                                                    | 0.82     | steel-stainless |
| Packing II (cross meshwork packing) | 0.285       | 1100                               | 1750                                                   | 0.86     | steel-stainless |
| Packing III (wave thread packing)   | 0.285       | 400                                | 350                                                    | 0.95     | steel-stainless |

Note: Diameter of wave thread packing of stainless steel was 0.285 mm, wavelength 3–5 mm and amplitude 2–3 mm.

(mm), and  $g$  represents the gravity acceleration which is  $9.8 \text{ m}\cdot\text{s}^{-2}$ . In rotor,  $n$  varies with  $r$  in the radial direction when  $\omega$  is constant. So, it is appropriate to use the average high gravity factor ( $\beta$ ) to depict the degree of high gravity field in this article.  $\beta$  is calculated as follows:

$$\beta = \frac{\int_{r_1}^{r_2} n \cdot 2\pi r dr}{\int_{r_1}^{r_2} 2\pi r dr} = \frac{2\omega^2 (r_1^2 + r_1 r_2 + r_2^2)}{3(r_1 + r_2)g} \quad (4)$$

where,  $r_1$  is the inner radius of rotor (mm), and  $r_2$  is the outer radius of rotor (mm).

Figures 2, 3, and 4 show the NTU as a function of  $\beta$  for RPB with three kinds of packings, given  $F$  of  $20 \text{ L}\cdot\text{h}^{-1}$  and  $R$  of 3. (1) As expected, NTU increases with  $\beta$  both for rectifying section and for stripping section. This is mainly because with the increase of  $\beta$ , the liquid film on the surface of packing becomes thinner, which results in larger contacting area for vapor and liquid, leading to better mass transfer efficiency of RPB. (2) The NTU of RPB2 (rectifying section) is larger than that of RPB1 (stripping section) during the operation, *i.e.*, the mass transfer efficiency of RPB2 is better than that of RPB1, which could be attributed to the difference of liquid film in the two RPBs. Since the thinner the liquid film in packing a RPB has, the better the mass transfer efficiency it has. In the experiment, RPB1 and RPB2 have the same structure and packing, and the spaces are big enough for the liquid and gas to flow through simultaneously. But, the quantity of liquid in RPB1 and RPB2 is dif-

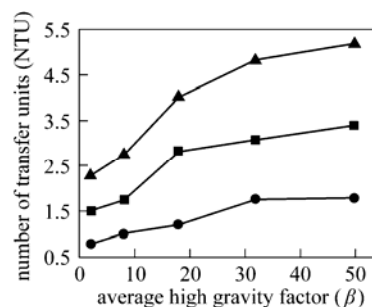


**Figure 4** The effect of  $\beta$  on NTU for packing III ( $F=20 \text{ L}\cdot\text{h}^{-1}$ ,  $R=3$ , at atmospheric pressure)  
● stripping section (RPB1); ■ rectifying section (RPB2); ▲ system

ferent. The quantity of liquid in RPB1 (stripping section) equals the quantity of feedstock plus that of reflux, whereas the quantity of liquid in RPB2 (rectifying section) only equals that of reflux, *i.e.*, the quantity of liquid in RPB1 is larger than that in RPB2. Therefore, with the same  $\beta$ , the liquid film on the surface of packing in RPB2 (rectifying section) is thinner than that in RPB1 (stripping section). (3) The maximum NTU of RPB2 is 3.4 in Fig. 4 and the minimum NTU of RPB1 is 0.52 in Fig. 2. So the HETP of RPB ranges from 7.35 mm to 48.07 mm given  $F$  of  $20 \text{ L}\cdot\text{h}^{-1}$ ,  $R$  of 3, and  $\beta$  varying from 1.99 to 49.81.

### 3.1.2 The effect of $R$ on NTU for RPB

Figures 5, 6, and 7 demonstrate the linear variation of NTU with  $R$  given  $\beta$  of 17.95 and  $F$  of  $24 \text{ L}\cdot\text{h}^{-1}$  for

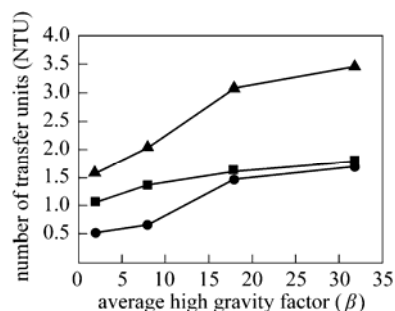


**Figure 2** The effect of  $\beta$  on NTU for packing I ( $F=20 \text{ L}\cdot\text{h}^{-1}$ ,  $R=3$ , at atmospheric pressure)  
● stripping section (RPB1); ■ rectifying section (RPB2); ▲ system

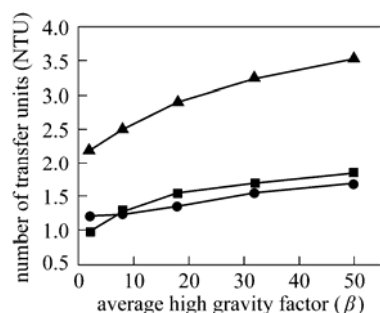


**Figure 3** The effect of  $\beta$  on NTU for packing II ( $F=20 \text{ L}\cdot\text{h}^{-1}$ ,  $R=3$ , at atmospheric pressure)  
● stripping section (RPB1); ■ rectifying section (RPB2); ▲ system

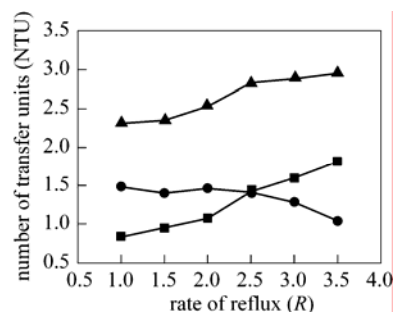


**Figure 5** The effect of  $R$  on NTU for packing I ( $\beta=17.95$ ,  $F=24 \text{ L}\cdot\text{h}^{-1}$ , at atmospheric pressure)  
● stripping section (RPB1); ■ rectifying section (RPB2); ▲ system

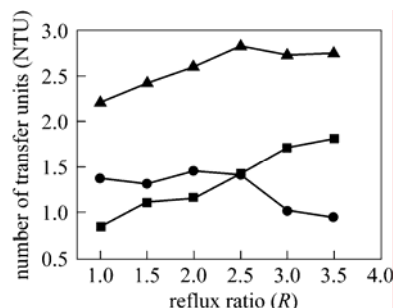


**Figure 6** The effect of  $R$  on NTU for packing II ( $\beta=17.95$ ,  $F=24 \text{ L}\cdot\text{h}^{-1}$ , at atmospheric pressure)  
● stripping section (RPB1); ■ rectifying section (RPB2); ▲ system

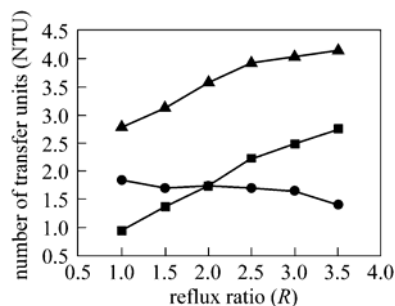


**Figure 7** The effect of  $R$  on NTU for packing III ( $\beta=17.95$ ,  $F=24 \text{ L}\cdot\text{h}^{-1}$ , at atmospheric pressure)  
 ● stripping section(RPB1); ■ rectifying section (RPB2);  
 ▲ system

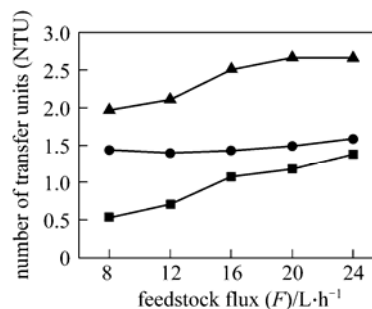


**Figure 8** The effect of  $F$  on NTU for packing II ( $\beta=49.81$ ,  $R=1$ , at atmospheric pressure)  
 ● stripping section(RPB1); ■ rectifying section(RPB2);  
 ▲ system

RPB with three kinds of packings. (1) On the basis of theoretical analysis, NTU is expected to increase with the reflux ratio. The data obtained has verified the expectations for the whole system but not for each section. NTU increases with  $R$  for rectifying section (RBP2) and decreases for stripping section (RBP1). This is mainly because the difference in  $R$  results in the difference in the quantity of liquid in each section, which leads to the difference in the thickness of liquid film in packings. In rectifying section, the quantity of liquid is much less than that in stripping section. Initially,  $R$  is little and the quantity of liquid is small, the contact area for vapor and liquid is small owing to the occurrence of dry surface area of packing, which leads to bad mass transfer efficiency. With the increase of  $R$ , the surface area of packing wetted becomes larger, leading to better mass transfer efficiency. However, in stripping section, the liquid covers all over the packing at first, *i.e.*, the packing is all wetted. With the increase of  $R$ , the liquid films in packing become thicker, which decreases the contacting area for vapor and liquid, resulting in worse mass transfer efficiency. (2) When  $R$  reaches 2.5, NTU of rectifying section equals that of stripping section for packing I, *i.e.*, the two RPBs have the same mass transfer efficiency. The intersection point for the two curves could be considered as an optimum operating condition in plant. (3) With the maximum NTU of 2.5 for RPB in Fig. 7 and the minimum NTU of 0.83 in Fig. 5, the HETP obtained for RPB varies from 10 mm to 30.12 mm given  $R$  ranging from 1 to 3.5,  $\beta$  of 17.95, and  $F$  of  $24 \text{ L}\cdot\text{h}^{-1}$ .

### 3.1.3 The effect of $F$ on NTU for RPB

Figures 8 and 9 display the relation of NTU and  $F$  given  $\beta$  of 49.81 and  $R$  of 1 for RPB with packing II and III. It is obvious that the NTU increases with  $F$  for both rectifying section and stripping section, especially for the former. This is mainly because circle velocity of liquid in packing increases with  $F$ . Larger circle velocity leads to larger surface area of packing wetted because the liquid distribution is not uniform in packing when  $F$  is small, so the contacting area for vapor and liquid becomes larger, and turbulence in vapor and liquid gets stronger. Hence, a better mass transfer efficiency of RPB is achieved. Simultaneously, the larger the  $F$  is, the larger the liquid drop in the inner of rotor becomes, which decreases the quantity of liquid discharged with vapor from RPB, thus improv-

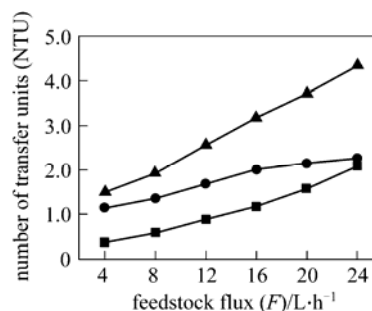


**Figure 9** The effect of  $F$  on NTU for packing III ( $\beta=49.81$ ,  $R=1$ , at atmospheric pressure)  
 ● stripping section(RPB1); ■ rectifying section(RPB2);  
 ▲ system

ing the mass transfer efficiency of RPB too. When the packing is totally wetted, the speed of NTU is increased slowly because of thicker film and shorter residual time of liquid in packing. The maximum NTU of RPB is 2.26 in Fig. 9. So the minimum HETP is 11.06 mm for packing III given  $\beta$  of 49.81,  $R$  of 1, and  $F$  of  $24 \text{ L}\cdot\text{h}^{-1}$ .

## 3.2 Mass transfer performance for three kinds of packings

### 3.2.1 The effect of $\beta$ on NTU for different packings

Figures 10 and 11 display the NTU and HETP as a function of factor  $\beta$  for the three kinds of packings of system given  $F$  of  $8 \text{ L}\cdot\text{h}^{-1}$  and  $R$  of 1. It is obvious that the NTU of each packing increases with  $\beta$  in Fig. 10

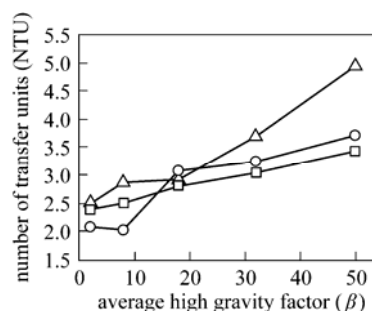


**Figure 10** The effect of  $\beta$  on NTU for packings ( $F=8 \text{ L}\cdot\text{h}^{-1}$ ,  $R=1$ , at atmospheric pressure)  
 ○ packing I; □ packing II; △ packing III

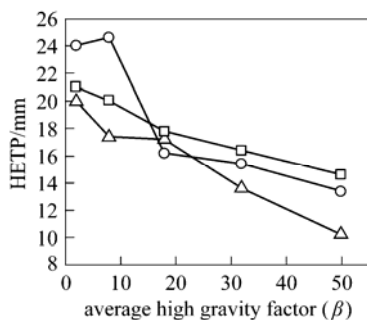


**Figure 11** The effect of  $\beta$  on HETP for packings ( $F=8 \text{ L}\cdot\text{h}^{-1}$ ,  $R=1$ , at atmospheric pressure)  
○ packing I; □ packing II; △ packing III

and the HETP decreases with  $\beta$  in Fig. 11. HETP of packing I, II, and III is 13.4 mm–24.6 mm, 14.6 mm–21.0 mm, and 10.2 mm–20.0 mm, respectively. Experimental data indicate that the packing III is the best among packings and  $\beta$  plays important role on mass transfer efficiency for RPB.

### 3.2.2 The effect of $R$ on NTU for different packings

The effects of  $R$  on NTU and HETP are shown in Figs. 12 and 13 for three kinds of packings of system given  $\beta$  of 17.95 and  $F$  of  $12 \text{ L}\cdot\text{h}^{-1}$ . The NTU of each packing increases with  $R$  in Fig. 12 and HETP decreases with  $R$  in Fig. 13. HETP ranges from 17.6 mm to 22.6 mm of packing I, from 17.6 mm to 21.2 mm of packing II, and from 12.8 mm to 19.0 mm of packing III. It is evitable that the mass transfer efficiency of packing III is better than those of packing I and II at the same value of reflux ratio.

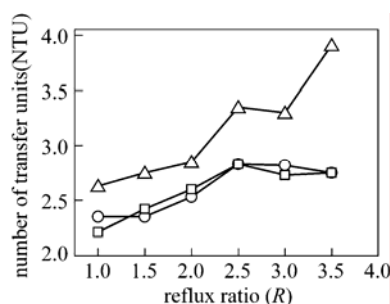


**Figure 12** The effect of  $R$  on NTU for packings ( $\beta=17.95$ ,  $F=12 \text{ L}\cdot\text{h}^{-1}$ , at atmospheric pressure)  
○ packing I; □ packing II; △ packing III

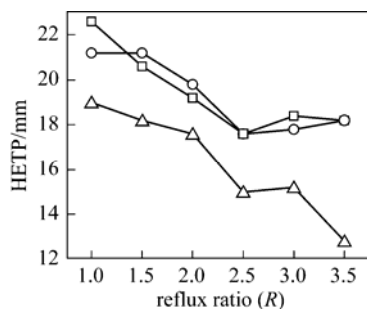


**Figure 13** The effect of  $R$  on HETP for packings ( $\beta=17.95$ ,  $F=12 \text{ L}\cdot\text{h}^{-1}$ , at atmospheric pressure)  
○ packing I; □ packing II; △ packing III

### 3.2.3 The effect of $F$ on NTU for different packings

Figures 14 and 15 display the variations of NTU and HETP with  $F$  for each packing of system given  $\beta$  of 17.95 and  $R$  of 1. The NTU increases with  $F$  for each curve seen in Fig. 14, and HETP decreases with  $F$  in Fig. 15. Also, the HETP of packing II is always larger than that of packing III, and smaller than that of packing I at the same value of  $F$ , implying the mass transfer efficiency of packing III is the best and that of packing I is the worst among the three packings. Experimental data reveals that HETP varies between 18.73 mm and 25.38 mm of packing I, 15.15 mm–25.00 mm of packing II, and 11.76 mm–23.58 mm of packing III.

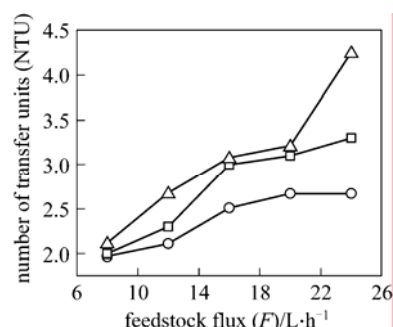


**Figure 14** The effect of  $F$  on NTU for packings ( $\beta=17.95$ ,  $R=1$ , at atmospheric pressure)  
○ packing I; □ packing II; △ packing III

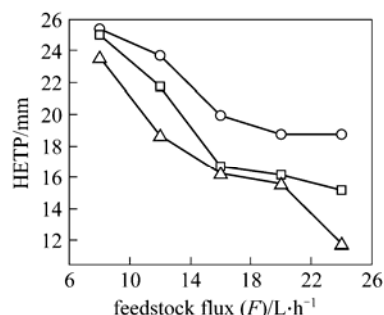


**Figure 15** The effect of  $F$  on HETP for packings ( $\beta=17.95$ ,  $R=1$ , at atmospheric pressure)  
○ packing I; □ packing II; △ packing III

## 4 MODEL DEVELOPMENT

In summary, the mass transfer efficiency of RPB is influenced by the average high gravity factor ( $\beta$ ), reflux ratio ( $R$ ), feedstock flux ( $F$ ), quantity of reflux ( $D$ ), concentrations of feedstock ( $x_F$ ) and that of production ( $x_D$ ). Based on the experimental data, semi-empirical correlation is developed initially as follows:

$$\text{NTU} = A\beta^a \left(\frac{D}{F}\right)^b R^c x_F^d x_D^e \quad (5)$$

where,  $A$ ,  $a$ ,  $b$ ,  $c$ ,  $d$ , and  $e$  are parameters needed to be calculated based on a great deal of experimental data.

Using the least square method, parameters could be calculated and models obtained for three packings could be expressed as follows:

**Packing I (corrugated disk packing)**

$$NTU = 1.26\beta^{0.0012}\left(\frac{D}{F}\right)^{0.125}R^{-0.257}x_F^{0.526}x_D^{1.916} \quad (6)$$

The accuracy of the model was checked using the square error analysis.  $F$  calculated was 75.75.  $F_{0.95}$  shown in Ref. [23] was 2.82.  $F$  is larger than  $F_{0.05}$ . The largest difference was 2.19% (calculated using MATLAB software). Mean difference was 0.72% (calculated using MATLAB software).

**Packing II (cross meshwork packing)**

$$NTU = 0.689\beta^{0.134}\left(\frac{D}{F}\right)^{-0.0079}R^{-0.170}x_F^{0.150}x_D^{1.507} \quad (7)$$

$F$  calculated was 53.53.  $F_{0.95}$  shown in Ref. [23] was 2.84.  $F$  is larger than  $F_{0.05}$ . The largest difference was 6.03% (calculated). Mean difference was 1.98% (calculated).

**Packing III (wave thread packing)**

$$NTU = 0.732\beta^{0.051}\left(\frac{D}{F}\right)^{-0.266}R^{-0.257}x_F^{-0.109}x_D^{1.046} \quad (8)$$

$F$  calculated was 53.53.  $F_{0.95}$  shown in Ref. [23] was 2.82.  $F$  is larger than  $F_{0.05}$ . The largest difference was 5.6% (calculated). Mean difference was 2.7% (calculated).

Figures 16, 17, and 18 show the comparison of the estimated and experimental NTU for packing I, II, and III. Apparently, the correlations are reasonable and could be used to predict the performance of RPB with experimental data.

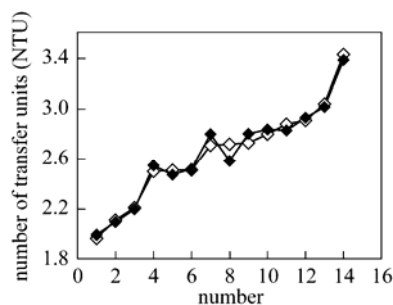


**Figure 16 Comparison of experimental and calculated NTU for packing I**

◇ calculated data; ◆ experimental data

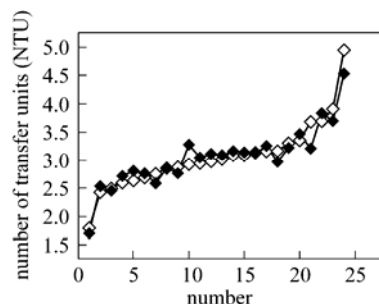


**Figure 17 Comparison of experimental and calculated NTU for packing II**

◇ calculated data; ◆ experimental data

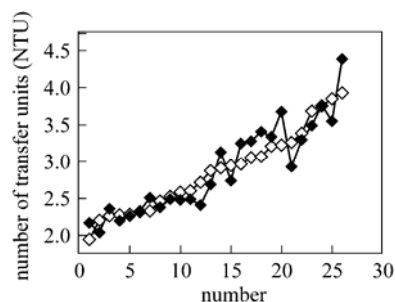


**Figure 18 Comparison of experimental and calculated NTU for packing III**

◇ calculated data; ◆ experimental data

In conclusion, The mass transfer efficiency of RPB is enhanced with the increase of average high gravity, reflux ratio, and feedstock flux in the scope of experimental operation. The correlations developed for the mass transfer of RPB, including an average high gravity factor ( $\beta$ ), reflux ratio( $R$ ), feedstock flux ( $F$ ), quantity of reflux ( $D$ ), concentrations of feedstock ( $x_F$ ) and production ( $x_D$ ), are reasonable. They could be used to predict the performance of RPB used in distillation. The packing plays an important role for RPB in distillation and should be considered when designing a scale up equipment. The wave thread packing of stainless steel had the best mass transfer efficiency among three packings in experiment.

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