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# Pressure drop behaviour and mass transfer properties of a high specific area random type packing in a narrow packed column

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*Abstract*: In this paper, the comprehensive experimental examinations are conducted to investigate the mass transfer properties of Dixon ring packing. The main aspect of this study is to investigate the characteristics of Dixon ring packing using a narrow packed column. Firstly, the mass transfer properties of the packing were investigated using distillation experiments at total reflux. Afterwards, the pervasive experiments were conducted to plot the generalized pressure drop correlation chart. Finally, the variation of height equivalent to a theoretical plate (HETP) was determined at total reflux operations for various vapour loading factors. Our findings showed that increasing the vapour loading factor up to 0.62 Pa<sup>0.5</sup> would eventually decrease the HETP. It was also shown that the further increase in the vapour loading factor results in a sudden increase in the HETP value. According to our findings, the selection of the optimum vapor loading factor would enhance the value of HETP up to more than 57 %.

*Keywords*: Dixon ring; packing characterization; distillation column; packed bed; narrow column; GPDC chart.

# INTRODUCTION

In recent years, the extensive studies have been conducted on light stable isotopes. Isotopes are different atomic types of a single element. Isotopes of different elements have applications in a variety of fields.<sup>1</sup> Take, for instance, the applications in the fields of medicine, pharmacology, biology and chemical engineering.<sup>2,3</sup> The light stable isotopes of elements such as carbon, oxygen and nitrogen have also applications in the study of human metabolism changes, the studies of molecular structure, non-invasive respiratory tests and Alzheimer tests.<sup>4</sup> Therefore, in recent years various researchers have extensively focused on



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the separation of light stable isotopes. With regard to molecular weight of the specified gaseous feed, various methods could be used for the separation of isotopes. Among these methods, cryogenic distillation is the only economic one for the large scale production of light stable isotopes.<sup>3</sup> The cryogenic distillation is a distillation process operated at temperatures below 120 K.

The difference between boiling points of isotopologues is the key isotope effect for the isotope separation by cryogenic distillation. This difference of boiling points between isotopologues is extremely small. Therefore, numerous stages are required for the isotope separation by cryogenic distillation. On the other hand, according to a rule of thumb, in small diameter packed columns, height equivalent to a theoretical plate (HETP) approximately equals the internal diameter of the column. Hence, the isotope separation by cryogenic distillation is usually conducted in relatively narrow columns (with diameters of about 40 mm and even smaller).<sup>1,5</sup> For an accurate design and an optimized operation of a packed distillation column, the knowledge about the characterizations of the packing must be complete and detailed. Among various characteristics of a packing, HETP and generalized pressure drop correlations (GPDC) are of great importance because HETP is often used to estimate the height of the distillation column and GPDC chart is commonly used to estimate the diameter or the capacity of the column. Thus, a significant number of studies could be cited in which the characteristics of a packing or a packed column was investigated.<sup>6-11</sup> However, most of these works could not be generalized to small diameter columns (5 cm and smaller) because they were conducted in relatively large diameter columns (larger than 15 cm). In other words, the results obtained from a small diameter column would be rigorously affected by wall effects, while it is not the case in a large diameter column. This could mainly be caused by the wall flow and the channelling phenomena, because at a constant liquid spraying density, decreasing the column diameter results in the increased wall flow.<sup>12</sup> Therefore, when a packing is to be used in a small diameter column (take, for instance, cryogenic distillation for isotope separation), it should better be characterized in a similar column.

Several studies so far focused on mass transfer properties or/and pressure drop behaviour of various types of packings. Yamanishi and Kinoshita<sup>13,14</sup> performed preliminary experimental studies on a small internal diameter cryogenic distillation column. They found that the over-all HETP was almost independent of the vapour flow rate. Olujic *et al.*<sup>15</sup> reported the results of Montz high capacity structured packings under total reflux distillation. They also presented the relationships between specific surface area, pressure drop, capacity, and separation efficiency. Del Carlo *et al.*<sup>16</sup> proposed a new mechanistic model to evaluate the mass transfer performance in columns equipped with structured packings. According to their findings, simple mechanistic models can be used with con-

fidence to describe the fluid dynamics within the packings. Jiangbo et al.<sup>17</sup> developed a three-dimensional two-phase flow model for simulating the hydrodynamics and mass-transfer properties in a typical representative unit of the structured packing. They showed that the values of HETP, estimated by the model, satisfactorily agreed with the experimental results. Li et al.<sup>3</sup> developed a new high surface area structured packing. They also designed and constructed the first <sup>13</sup>C separation pilot-scale plant using the structured packing. Moreover, they developed the fluid mechanical and mass transfer empirical equations according to their new structured packing. Bessou et al.<sup>18</sup> performed several experiments on a new packing using a 150 mm internal diameter column. They compared the properties of the packing with those of packings generally used in distillation and absorption. Olujic *et al.*<sup>19</sup> presented the results of the total reflux distillation for various packings using a 590 mm internal diameter column. Their results suggested that Delft model closely estimates the pressure drop and the efficiency of the packing species. Wang et al.<sup>20</sup> characterized a packing type which is widely used in post-combustion  $CO_2$  capture. They reported the mass transfer properties of the packing. Olujic *et al.*<sup>21</sup> employed a 1.22 m internal diameter column for total reflux distillation experiments at different pressures. Their findings showed that approximately the same efficiency values could be obtained at vacuum and above atmospheric pressures. Weng et al.<sup>6</sup> characterized a new spherical metal mesh random packing (SMMP). Their results indicated the potential of developing high-efficiency and energy-saving SMMP-based equipment.

Although many research works could be cited in this field, to the best of our knowledge no remarkable job is performed on the characterization of the mass transfer properties of random packings in a narrow packed column. The main objective of the present work is to characterize the mass transfer properties and pressure drop behaviour of the Dixon ring packing in a small internal diameter column. Note that for sizing/rating of a distillation column using a specified packing, two main characteristics of the column should be determined. The diameter of the column is the first, and the second one is the height of the packed bed. Sizing/rating of the diameter of the column is performed using GPDC chart which is commonly plotted using the results of pressure drop experiments. On the other hand, the determination of height of the bed is accomplished using the data of HETP. In this research work, first, five millimetres Dixon ring packing is constructed. Then a narrow distillation column is employed to investigate the mass transfer properties of the packing under total reflux conditions. Afterwards, the column is used to define the dry and wet pressure drop chart. The pressure drop chart is then used to develop the GPDC chart for the packing at various vapour and liquid loadings. Finally, HETP is defined versus vapour loading factor and the optimal operating value for the vapour loading factor is determined.

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# EXPERIMENTAL

#### Packing type

Five millimetres Dixon ring packing was investigated. This type of random packing is famous for its large specific surface area accompanied with the small pressure drop. Structural characteristics of the investigated packing are presented in Table I.

TABLE I. Structural characteristics of the 5 mm Dixon ring packing

Material	Size mm <sup>2</sup>	Specific surface area, m <sup>2</sup> m <sup>-3</sup>	Density kg m <sup>-3</sup>	Void fraction %	Packing factor m <sup>-1</sup>
SS-316 Wire netting 0.40×60 mesh	5×5	1570	430	94.9	1743

#### Experimental setup

A schematic view of the experimental setup is shown in Fig. 1. The main part of the setup is a randomly packed distillation column. The setup is also supplied by the supplementary devices for the manipulation of the distillation process. The experimental column is a modular narrow column with an internal diameter of 39.5 mm. The height of each module is 25 cm. On the top of every module a funnel type liquid distributor is devised. The liquid redistributor is depicted in Fig. 2. It should be noticed that the spacing between two adjacent distributors was 25 cm in order to prevent the channelling phenomena and also to reduce the wall flow. Dixon ring packing was used to pack the column. This type of random packing is commonly used in narrow columns.<sup>13,14</sup>



Fig. 1. Schematic view of the experimental setup.

#### Experimental procedure

Two sets of experiments were conducted. The first set of experiments was used to estimate the mass transfer properties of the packing. In the second set of experiments, the pressure drop tests were performed.



Fig. 2. Liquid redistributer.

#### Rectifying tests

A mixture of 45 mole percent methylcyclohexane in n-heptane was used as the feed in the rectification experiments.<sup>22</sup> All of the rectifying experiments were conducted under total reflux condition.

To start the rectification, initially two litres of the feed was charged into the reboiler vessel. Then, the Condenser was started to refrigerate the column. Afterwards, the reboiler was started with a specified heating power which should be approximately 30–40 % lower than the operating power of reboiler. It should be noted that the operating power of a reboiler is specified regarding the demanded vapour loading factor. Vapour loading factor can be calculated using the Eq. (1):

Vapor loading factor 
$$(F_factor) = U_g \sqrt{\rho_g}$$
 (1)

where  $F_{factor}$  is the vapor loading factor,  $U_g$  and  $\rho_g$  are the superficial velocity and the density of the vapour, respectively.

#### Pressure drop tests

Pressure drop experiments were conducted using water and air as the liquid and vapour phases, respectively. To perform the pressure drop tests the condenser and the reboiler of the experimental system were modified. A polypropylene vessel was devised instead of the condenser to supply water with a constant flow rate to the column. In addition, a simple method was used to maintain the constant water flow rate. Details of the water flow maintenance system are as follows:

As shown in Fig. 3, to supply water with a constant flow rate to the column, water was pumped from vessel 1 to vessel 2 on which a valve and a water flow meter were devised. Then, the desired flow rate to the column was regulated using the flow meter 3 and the extra water, discharged to vessel 2, was gravitationally returned to vessel 1 through valve 4. Therefore, this process simply leaded to a constant liquid level in vessel 2, which in case leaded to constant water flow through the flow meter 3.

Other experimental details are given in Supplementary material to this paper.

### RESULTS AND DISCUSSION

### GPDC development

The pressure drop data of a packing is crucially important when designing a distillation process. To accurately design and control the distillation process, the





Fig. 3. The setup used for regulation of flow rate of liquid.

GPDC chart of the packing should be developed. The question is what should be the range of the liquid spraying density and the gas loading factor?

Relative volatility is one of the main criteria of the difficulty of a distillation process. The smaller the relative volatility is, the more troublesome the separation process would be. On the other hand, increasing the pressure would decrease the relative volatility.<sup>23</sup> Thus, any activity which leads to the increased pressure drop must be avoided. Hence, cryogenic distillation plants used for isotope separation are usually operated using gas loading factors which are relatively small.<sup>13,14</sup> Therefore, the GPDC chart is plotted for relatively small gas loading factors and various liquid spraying densities. Fig. 4 shows the variations of pressure drop *versus* volume flow rate of air for different liquid spraying densities.





After drawing the complete pressure drop chart, the GPDC chart could be plotted. To plot the GPDC chart, the values of X (the value of abscissa) and Y (the value of ordinate) should be calculated for constant pressure drop curves:

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$$X = \frac{L_{\rm P} \rho_{\rm v}^{0.5}}{G_{\rm P} \rho_{\rm l}^{0.5}} \text{ and } Y = \frac{G_{\rm P}^2 \mu_{\rm L}^{0.1} F_{\rm P} (\rho_{\rm w} / \rho_{\rm l})^{0.5}}{32.17 \, \rho_{\rm v} (\rho_{\rm l} - \rho_{\rm v})}$$

in which  $\rho_v$  is the density of vapour phase,  $\rho_l$  is the density of liquid phase,  $\rho_w$  is the density of water,  $F_P$  is the packing factor and  $G_P$  and  $L_P$  are the mass flow of vapour and liquid for a unit cross sectional area of the column, respectively. The term of  $(\rho_w / \rho_l)^{0.5}$  was introduced and used in the expression of Y due

The term of  $(\rho_w / \rho_l)^{0.3}$  was introduced and used in the expression of Y due to the omission of the operational liquid density effect. In other words, the aforementioned term was utilized in order to plot a GPDC in which the effect of the specific operational liquid in the column is removed.<sup>24</sup>

To do so, the following procedure should be accomplished:

1. A square polynomial curve is fitted on the data points of pressure drop versus volume flow rate of vapour (Fig. 4). This fitted equation is used to estimate the fluctuations of pressure drop as a function of a variation in the volume flow rate of vapour. The obtained results for the fitted polynomials are displayed in Table II.

2. A value of the pressure drop is selected as a base value for calculations of the GPDC chart. This base value is the value of one of the constant pressure drop curves which finally appears in the GPDC chart.

3. The values of  $L_P$  corresponding to the investigated values of spraying density of liquid are simply calculated. Afterwards, the values of  $G_P$  are calculated for the specified vapour loading factors using the fitted equation in step 1.

4. By the substitution of the acquired results of the previous steps, the values of X and Y are calculated.

5. When step 4 is completed, a set of X and Y is obtained for each base value of pressure drop at every spraying density of liquid.

6. Finally, the values of *X*, *Y* and the base pressure drop are used to plot the GPDC chart.

The GPDC chart is depicted in Fig. 5.

TABLE II. The data of the fitted polynomials of pressure drop versus vapor loading factor for constant liquid loadings; *A*, *B* and *C* are the constants of the fitted equation with the form of  $Q = A\Delta P^2 + B\Delta P + C$  in which *Q* is the volume flow rate of vapor in L s<sup>-1</sup> and  $\Delta P$  is the pressure drop in mbar m<sup>-1</sup>; range of vapor loading factor: 0.2–1.3 m<sup>3</sup> m<sup>-2</sup> s<sup>-1</sup>

Loading factor of liquid	Range of pressure drop	A	В	С
mms	mbar m <sup>-</sup>			
0.0006	1-11	-0.0031	0.1494	0.1152
0.001	1–12	-0.0032	0.141	0.1083
0.002	1–13	-0.0027	0.1305	0.1179
0.005	1–16	-0.0026	0.1231	0.1349
0.0075	1-18	-0.0022	0.1135	0.1137

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Fig. 5. The generalized pressure drop correlation for 5 mm Dixon ring packing in a column with an internal diameter of 39.5 mm. The values of pressure drop for the constant pressure drop curves are reported in two dimensions: inches of water per foot of packing height and mbar per m of packing (in parentheses).

The GPDC chart contains the most essential and the most valuable data about a packing. This chart could be simply used to size the column. Once the GPDC chart is developed, the optimal diameter of the column for a specified value of pressure drop could be estimated. Commonly a trial and error procedure is employed to design the diameter of a distillation column for a specified pressure drop:

1. First, the equilibrium stage modelling is used to simulate the rectification process for the specified separation criteria (concentration or recovery).

2. The values of internal flow rates of liquid and vapour and also the values of density are employed to calculate the abscissa of the chart  $(X = L_P \rho_v^{0.5} / (G_P \rho_l^{0.5}))$ . Note that  $L_P$  and  $G_P$  are respectively loadings of liquid and vapor and are not specified in the results of equilibrium modeling. Instead, the internal flow rates of liquid and vapour are specified. Although the values of loading do not equal to that of flow rates, the ratio  $L_P/G_P$  exactly equals the ratio internal liquid flow/internal vapour flow.

3. The desired pressure drop is calculated using a specified criterion. For instance, a pressure drop corresponding to 80 % of flooding pressure drop could be used.

4. Using the value of abscissa from step 2 and the desired pressure drop from step 3, the corresponding constant pressure drop curve is considered to evaluate the ordinate of the chart:

$$Y = G_{\rm P}^2 \mu_{\rm L}^{0.1} F_{\rm P} (\rho_{\rm w} / \rho_{\rm l})^{0.5} / (32.17 \, \rho_{\rm v} (\rho_{\rm l} - \rho_{\rm v}))$$

5. Substituting the parameters of Y and solving the equation for  $G_P$ , the value of  $G_P$  is defined.

6. Finally,  $G_P$  from step 5 is compared to the value of vapour flow rate from step 1 to obtain a value of cross sectional area. The cross sectional area is in turn used to calculate the diameter of the column.

7. The procedure of steps 2-6 is repeated for a new value as the base pressure drop.

Hence, using the developed GPDC, rating or sizing of a distillation column packed with Dixon ring packing could be accurately performed.

# Rectification process

Operation of rectification processes in narrow columns is of great importance in the isotope separation by distillation. On the other hand, due to relatively small separation factors in isotope separation processes, hundreds and actually thousands of ideal stages are needed to achieve an acceptable enrichment. Hence, a precise estimation of the HETP value is a must. Fig. 6 plots the results of HETP as a function of the power of reboiler at the total reflux operation. Note that the variation of the power of reboiler results in different values of vapour loading factor, hence, the HETP is plotted against both the reboiler duty and the vapour loading factor.

The results in Fig. 6 show that increasing the vapour loading factor from 0.35 to 0.62 (points A to C) would decrease the HETP from almost 9.75 to 6.2 cm. It can also be observed that the further increase in vapour loading factor beyond 0.62 (points C to D) would increase the HETP to approximately 9 cm. Hence, assuming an efficiency of 100 %, if the vapour loading factor is to be controlled about 0.35, every 9.75 cm of the packed section works as a single stage. On the other hand, and again with an efficiency of 100 %, at a vapour loading factor of 0.62, every 6.2 cm of the packed section works as a single stage. Suppose only 10 stages are required for the specified separation, then the demanded height of the packed section in the first column and the second one would approximately be 97.5 and 62 cm, respectively. This means that the efficiency of the same column at different operating conditions could fluctuate even more than 50 %. It should be taken into account that one of the main applications of narrow distillation columns is the separation of light stable isotopes. As discussed earlier, relative volatility of stable isotopes of light elements is intensively small. On the other hand, controlling a distillation column at the point of maximum separation efficiency (point C) is drastically difficult. At the point of maximum separation efficiency, the flow rate of vapour becomes relatively large so that the column gets extremely close to the flooding point. Therefore, every small fluctuation in the system could lead to a failure in controlling strategies which in case results in flooding. Hence, the distillation columns are commonly

operated in the vapour loading factors corresponding to 80 % of vapour loading factor of the flooding point. This approximately corresponds to the point B in Fig. 6.



Fig. 6. Fluctuations of HETP as a result of variations of vapour loading factor at total reflux operation.

# CONCLUSION

In this paper, the extensive experimental investigations were performed on 5 mm Dixon ring packing to characterize its mass transfer characteristics. This study tries to examine the pressure drop behaviour of the packing species. All of the experimental tests were conducted in a small internal diameter column with an internal diameter of 39.5 mm. In order to decrease wall effects and especially wall flow, the liquid was redistributed once a 25 cm of the column. Wet pressure drop was extensively investigated at various vapour loading factors in the range of 0.2–1.4 Pa<sup>0.5</sup> for a variety of liquid spraying densities in the range of 0.0006–  $-0.0075 \text{ m}^3 \text{ m}^{-2} \text{ s}^{-1}$ . Moreover, the mass transfer properties were characterized by the experimental examinations on the value of height equivalent to a theoretical plate (HETP). Afterwards, the variations of HETP were investigated versus various vapour loading factors in the range of 0.35-0.66 Pa<sup>0.5</sup> at total reflux distillations. The results of the wet pressure drop experiments were used to plot the generalized pressure drop correlation (GPDC) chart for pressure drop values in the range of 0.124-1.612'' of water/foot of packing  $(1-13 \text{ mbar m}^{-1})$ . Then, the procedure of sizing of a distillation column with regard to the developed GPDC chart was thoroughly described. The results of the rectifying tests were used to plot the value of HETP as a function of the vapour loading factor. It was concluded that increasing the value of the vapour loading factor from 0.35 to 0.62 Pa<sup>0.5</sup> decreases the HETP value from 9.75 to 6.2 cm. It was also found that the further increase in vapour loading factor beyond 0.62 Pa<sup>0.5</sup> would increase the value of HETP up to almost 9 cm for the vapour loading factor of 0.66 Pa<sup>0.5</sup>.

Finally, it was concluded that applying the 5-mm Dixon ring packing in distillation experiments using a narrow column should be conducted in the vapour loading factors of almost 0.58–0.62 Pa<sup>0.5</sup>, in order to achieve an optimum value of HETP.

### SUPPLEMENTARY MATERIAL

Additional experimental data are available electronically at the pages of journal website: http://www.shd.org.rs/JSCS/, or from the corresponding author on request.

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#### ИЗВОД

# ПАД ПРИТИСКА И ПРЕНОС МАСЕ У КОЛОНАМА МАЛИХ ПРЕЧНИКА СА НЕУРЕЂЕНИМ ПАКОВАЊЕМ ВИСОКЕ СПЕЦИФИЧНЕ ПОВРШИНЕ

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У овом раду су приказани резултати опсежних експерименталних испитивања која су извршена у циљу испитивања карактеристика паковања у облику Диксонових прстенова (Dixon ring) у погледу преноса масе. Основни аспецт овог истраживања је да се испитају карактеристике паковања у облику Диксонових прстенова у пакованим колонама малих пречника. Својства у погледу преноса масе су прво испитивана коришћењем дестилационих експеримената при тоталном рефлуксу. Затим су вршени первазивни експерименти да би се добио дијаграм генерализованог пада притиска. На крају, одређена је промена висине еквивалентне теоријском поду (HETP) при тоталном рефлуксу, при промени фактора оптерећења паре. Резултати су показали да пораст фактора оптерећења паре до 0,62 Ра<sup>0,5</sup> смањује HETP. Такође је показано да даљи пораст фактора оптерећења паре резултује наглим порастом вредности HETP. На основу резултата, избором оптималне вредности фактора оптерећења паре, вредност HETP се може повећати за више од 57.

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