APPARATUS FOR MASS AND HEAT TRANSFER IN FLOWING LIQUID FILM

THE EFFECT OF PRESSURE ON EFFICIENCY OF FLAT PACKING IN VACUUM RECTIFICATION

M.Endršt, J.Červenka and V.Kolář

Institute of Chemical Process Fundamentals, Czechoslovak Academy of Sciences, 165 02 Prague - Suchdol

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The effect of pressure on efficiency of flat packing is studied in vacuum rectification for the chlorobenzene–ethylbenzene system. The internals are made of expanded metal sheets. Number of theoretical plates and pressure drop are measured. Results are compared with data given in literature.

Pressure drop between the reboiler and the head of column is of considerable importance in vacuum rectification and it also depends on the design of column internals. Standard packings of industrial columns usually have large pressure drop so that the vacuum column must be divided into several sections which negatively affects the economy of the process. For these reasons special types of column internals were designed for vacuum distillation characterized by low pressure drop often paid for by the loss in efficiency or complex design.

As the packing made of expanded metal sheets had in rectification and absorption at atmospheric pressure very good efficiency and very low pressure drop even at high through-puts, it was possible to expect that it would be suitable for vacuum rectification as well.

Here experimental efficiencies and pressure drops obtained in columns with this type of internals are summarized for various pressures and various vapour loads at total reflux for one system.

The vapour density and the vapour flow rate in the column are decreasing with decreasing pressure. In packed columns which are mostly used industrially at low liquid loads the irregularity of liquid flow down the packing increases which results in insufficient wetting of the packing and, consequently, in decrease of interfacial area and over-all efficiency.

In vacuum distillation, Billet[1,2] has used four binary systems: benzene–ethylene chloride, benzene–toluene, ethylene chloride–toluene and ethyl alcohol–water. The main attention has been paid to the efficiency of internals, pressure drops and column loads. The measurements were performed on Raschig rings 8 · 8 mm and 25 · 25 mm, packing height was 1 000 mm and pressure was altered in the range from 15 to 760 Torr. Number of theoretical plates per meter of height

of internals at the highest through-puts is expressed by the relation

$$\mathrm{NTP}/H = 0{\cdot}13\,\xi\,, \tag{1}$$

where ξ is defined by the known relation for pressure drop

$$\Delta P/H = \xi\varrho(u^2/2)(1/d)\,. \tag{2}$$

The vapour flow rate corresponding to loads at which the maximum number of theoretical plates can be calculated from the relation

$$u_m = K(\varrho_L/\varrho_V)^{1/2}\,. \tag{3}$$

where K is the function of the type of packing.

For the dependence of the number of theoretical plates on vapour load a rapid increase in vicinity of the flooding point is typical which can be in atmospheric distillation up to 50% greater than the mean value. With decreasing pressure, the difference between the maximum and mean value of the number of theoretical plates decreases.

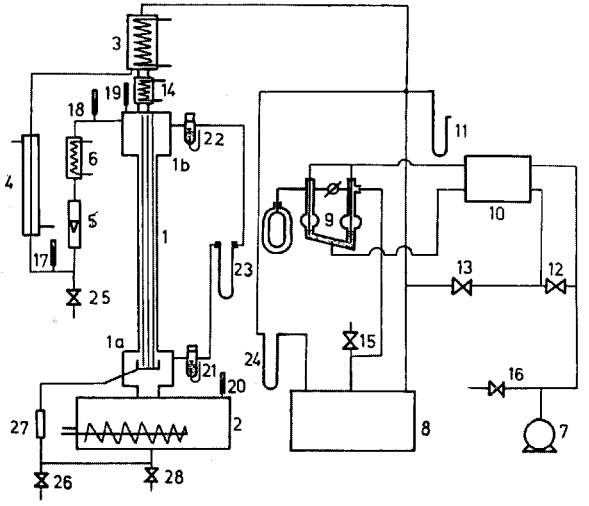

FIG. 1

Experimental Apparatus

1 Column, 2 reboiler, 3 condenser, 4 cooler of distillate, 5 rotameter, 6 preheater of reflux, 7 oil vacuum pump, 8 vacuum reservoir, 9 manostat, 10 vacuum controller, 11 mercury manometer, 12 recycle for initial operation of the column, 13 vacuum valve, 14 superheating of vapours, 15, 16 air operating valve, 17, 18, 19, 20 thermometers, 21, 22 air inlet into the impulse piping and to pressure taps, 23 pressure taps for measuring the pressure drop over the column, 24 manometer.

EXPERIMENTAL

The distillation apparatus is drawn schematically in Fig. 1. Vapours from the reboiler 2 are passing through the column 1 equipped with the internals made of expanded metal and condensate in the condenser 3. Distillate after cooling in the cooler 4 is passed through the rotameter 5 and before the inlet into the column head is heated in the heat exchanger 6 to the temperature by several degrees smaller than the temperature of vapours in the column head. The reboiler 2 is heated by the resistance heater consisting of three sections with the power input 2750 W each from which two sections are independently controllable. The column 1 is a pipe made of technical glass with ID 50 mm, 1500 mm long. The bottom and top end of the pipe is mounted into a larger pipe (1a, b). In the upper larger pipe is situated the liquid distributor into which the reflux from the heater is fed. Liquid from the internals is collected in the collector and from there supplied into the reboiler. The column internals were formed by a bundle of parallel sheets with the distance of plates 4·5 mm. The expanded metal sheets with the size of holes 10 . 5 mm were used. As the source of vacuum the rotary oil vacuum pump 7 was used. The condenser was connected with the vacuum pump through the steel vacuum chamber 8 with the ID 350 mm and 900 mm long. Vacuum was controlled by the manostat 9 filled with mercury. Pressure drop in the column was measured by two taps situated in metal pipes in the column head and bottom and was measured by a micromanometer 23. A small amount of air was sucked into the impuls pipe through the bubbled vessel 21, 22 to prevent flow of vapours into the pressure taps.

Measured quantities were: the amount of distillate, pressure drop, composition of distillate and of the residue and the temperature of vapours at the inlet and outlet from the column, the temperature of the reflux before the inlet into the column and in front of the rotameter. Distillate and residue were sampled into glass vessels 25, 26 which were connected to the source of vacuum.

System used and analytical methods. For this study, the system chlorobenzene–ethylbenzene was used which is recommended as the testing system for vacuum rectification by the European Federation of Chemical Engineers[3–5] and for which the needed physico-chemical data are available. The mixture behaves according to the Raoult's law. For determination of the vapour–liquid equilibria, the Antoine equation was used with the constants given in literature[5]. Analysis of the samples was made by measuring the density of the mixture by the digital densitometer DMA-02 of the Paar Co. Maintaining the temperature in the range 20·00 ± 0·02 C the molar ratio can be used with an accuracy 0·002.

Evaluation of data. Number of theoretical plates and pressure drop per unit length and per unit of theoretical plate in dependence on the *F*-factor $F = u \,.\, \varrho_V^{1/2}$ were determined.

Number of theoretical plates was determined by the McCabe–Thiele method and the number of transfer units in the vapour phase

$$N_{OG} = \int_{y_w}^{y_H} \frac{dy}{y^* - y}$$

was calculated by the Simpson's method so that the distance between y_w and y_H was divided into 100 parts.

RESULTS AND DISCUSSION

The effect of pressure on efficiency of distillation columns given in literature is often contradictory. Majority of authors agree that with decreasing pressure the efficiency

of column internals decreases, the Russian authors in their monography[6] are giving examples in which decrease in efficiency has not been observed.

Results of our experiments are given in Fig. 2 where the efficiency of the packing and liquid load are plotted in dependence on pressure. For the same system Zogg presents on the Sulzer packing, which is for its large efficiency especially advantageous for vacuum rectification, the decrease in efficiency down by 50% at the change of pressure from 300 Torr to 20 Torr. As not enough additional data are available in literature which could enable comparison of the packing efficiency with pressure for the chlorobenzene–ethylbenzene system, several systems have been selected for comparison which are given by Billet[1,2]. Data for expanded metal correspond to about 80% flooding (taken for $\Delta P/H = 30$ mm water column), for other internals values of maximum efficiency or of maximum vapour loads are plotted.

An important observation is that the decrease in efficiency on the internals made of expanded metal sheets is very small. For a 30-fold decrease in pressure, the efficiency decreases at distillation of the considered mixture by 20%. The compared results of other authors measured on the packed and plate internals show that efficiency decreases considerably faster with decreasing pressure.

A typical dependence of efficiency and of pressure drop on the rate factor F which has been obtained in our experiments is plotted in Fig. 3. The dependence has a similar character for other pressures. Increasing efficiency at low vapour loads can be explained by a longer contact time. This effect is probably prevailing over the deterior-

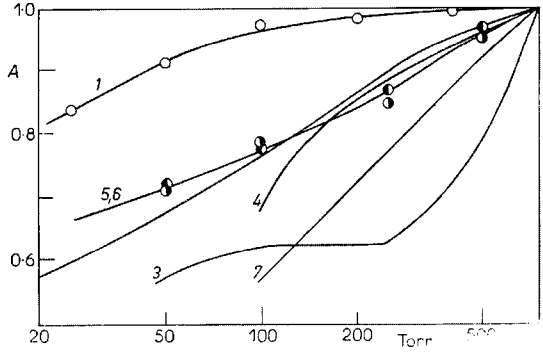

FIG. 2

Efficiency in Dependence on Operating Pressure

A Ratio of number of theoretical plates per unit height at operating pressure to the number of theoretical plates at atmospheric pressure. *1* chlorobenzene–ethylbenzene, expanded metal sheets with mesh size 10 . 5 mm, *2* ethylene chloride–toluene, Raschig rings 8 . 8 mm, $x_w = 5\%$, *3* ethylene chloride–toluene, Raschig rings 8 × 8 mm, $x_w = 3\%$, *4* benzene–toluene, Raschig rings 8 × 8 mm, *5* ethanol–water. ◑ Raschig rings 25 × 25 mm, *6* ethanol water, ◐ sieve plate, *7* benzene–ethylene chloride, Raschig rings 8 × 8 mm.

ated wetting of the surface which is in this region quite irregular. At large vapour loads the inbuilt interfacial area is better used especially in vicinity of the loading point. Increase in efficiency in this region is not so impressive as with packed columns.

Pressure drop per unit of column length in dependence on the rate of vapour phase is plotted in Fig. 4. On this curves the breaks corresponding to the beginning of liquid loading are remarkable. With decreasing pressure the slope of the straight line for the region of liquid loading increaes. The second break appearing for pressures 400 mm Hg and 760 mm Hg at low vapour rates is perhaps the result of considerable heat loss, as the difference between the temperature of the vapours and the temperature of the surrounding is at higher pressures considerable. Some pressure drop can be measured at low heating intensity even for a good insulation though no more vapours enter the condenser due, their condensation on the walls.

Finally, it can be concluded that in agreement with the results obtained by various authors the efficiency of the expanded metal sheet packing decreases with decreasing pressure though the decrease is very slow. This fact, good efficiency, high through-puts and a very low pressure drop are the main advantages due to which are these internals very attractive for practical application.

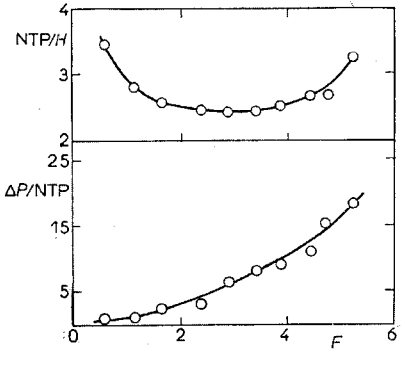

FIG. 3

Typical Shape of Experimental Number of Theoretical Plates Per Unit of Height (m^{-1}) and Pressure Drop Over the Theoretical Plate (mm water column) in Dependence on the Velocity Factor F ($kg^{1/2}\ s^{-1}\ m^{-1/2}$)

System chlorobenzene–ethylbenzene, P = = 50 Torr.

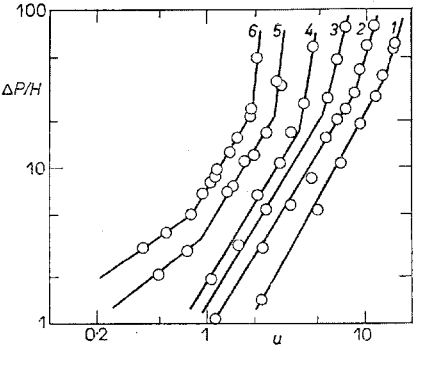

FIG. 4

Pressure Drop per Unit of Height (mm wc/m) in Dependence on Velocity of Vapours in the Column (m/s) for System Chlorobenzene–Ethylbenzene

P (Torr) *1* 25; *2* 50; *3* 100; *4* 200; *5* 400; *6* 740.

LIST OF SYMBOLS

d diameter of packing
F velocity factor
H column height
NTP number of theoretical plates
P pressure
ΔP pressure drop
u vapour velocity
y molar ratio in vapour phase
ϱ density
ξ friction factor

Subscripts

A, B binary components
H column head
L liquid phase
V vapour phase
m maximum
w backflow from the column into the reboiler
x equilibrium

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