

# AN EQUIPMENT FOR MASS AND HEAT TRANSFER TO A FILM OF LIQUID FLOWING DOWN A PLANE SURFACE. IV.\*

## THE EFFICIENCY OF THE FLAT PACKING FOR RECTIFICATION OF THE METHANOL-WATER MIXTURE

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The paper deals with experimental determination of the height of a transfer unit for rectification of the methanol-water mixture at total reflux, various mixture compositions and flow rates in a column packed with strips of expanded metal. The HTU obtained are compared with those found on other types of equipment and the same system.

In the preceding communications of this series we have dealt with the efficiency of the flat packing made of expanded metal for absorption processes. The present paper summarizes the results of an experimental investigation of the rectification efficiency of the same packing for the methanol-water binary. Similarly as in the preceding communication<sup>1</sup>, the main aim of this paper is to compare our results with those obtained on other types of equipment and the same system.

Adolphi and coworkers<sup>2</sup> rectified the methanol-water mixture on packings consisting of flat sheets. As variables they chose the spacing of the sheets (7; 12; 24 mm) and the velocity of vapour (0.5 to 5 m/s). From their results it is apparent that the number of theoretical plates per unit height, NTP, slightly decreases with the vapour velocity for all three geometric arrangements. No unambiguous effect of the sheet spacing was detected. At the velocity 2 m/s and 7 mm spacing the authors achieved  $NTP = 0.7 \text{ m}^{-1}$ ; at 24 mm spacing  $NTP = 0.8 \text{ m}^{-1}$ . Bancroft and coworkers<sup>3</sup> rectified the  $\text{H}_2\text{O}$ -HDO and ethanol-2-propanol mixtures and their height of a transfer unit varied between 2 to 6 ft and 6.5 to 13 ft respectively. In a long-term vacuum rectification of the cyclohexanone-cyclohexanol mixture in a 150 mm tower 3400 mm long Olevskii and coworkers<sup>4</sup> achieved the height of a transfer unit 0.8 to 1.3 m. Maljusov and coworkers<sup>5</sup> found at rectification of water the height of a transfer unit to be independent of the density of wetting and equal to 0.9 m at the temperature ranging between 40–60°C, or 0.81 m at 80–100°C, *i.e.* a decrease with the temperature. Finally, Kan and coworkers<sup>6</sup> found that HTU for rectification of the methanol-water system increases with decreasing concentration of methanol (20% methanol mixture HTU = 1–0.7 m; 60% methanol mixture HTU = 0.6 m). For 20% solution of methanol the height of a transfer unit decreases with increasing vapour Reynolds number.

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From the above review it is apparent that the rectification efficiency of flat packings is markedly inferior to that of the common types of plate and packed columns.

## EXPERIMENTAL

**Apparatus.** A block diagram of experimental set-up is shown in Fig. 1. The column 1 was a glass tube 50 mm in inner diameter. The packing was made of strips of expanded metal forming two 900 mm sections (2a, 2b) equipped on top with liquid distributors (3a, 3b). The boiler 4 was a 150 mm cylindrical vessel 1000 mm long. The heat was supplied into the boiler by two electric coils consisting each of three 3000 W independently controllable sections. One of the section was powered by an adjustable-ratio autotransformer for continuous control of the power input. The boiler was equipped with a filler, an inlet branch for liquid returning from the column, an outlet branch for vapours, and a level gauge. The vapours were entering the bottom part of the column provided with a thermometer 5 and left the column *via* thermometer 6 into a condenser 7 to be totally condensed. The condensate was returned *via* a thermostat 8 kept at  $20 \pm$

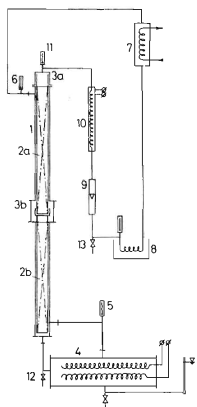


FIG. 1

A Block Diagram of Experimental Set-Up  
 1 Column jacket, 2a, 2b strips of expanded metal, 3a, 3b liquid distributor, 4 boiler, 5 thermometer, 6 thermometer, 7 condenser, 8 thermostat, 9 rotameter, 10 reflux preheater, 11 thermometer, 12 sampling tap, 13 sampling tap.

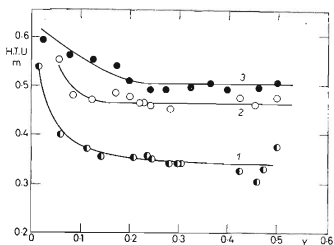


FIG. 2

A Plot of HTU against Mole Fraction of Methanol in Vapour Entering the Column  
 Expanded metal  $16 \times 5.5$  mm mesh, 12.5 mm spacing 1 3460–4 560  $\text{kg/m}^2 \text{ h}$ , 2 7420 to 8 520  $\text{kg/m}^2 \text{ h}$ , 3 9600–12 380  $\text{kg/m}^2 \text{ h}$ .

$\pm 0.1^\circ\text{C}$ , a rotameter 9 and a preheater 10 as a reflux into the column. The temperature of the reflux was measured at the column inlet by a thermometer 11. The bottom product in the boiler and the distillate were sampled by taps 12 and 13. The whole column was insulated by polystyrene foam. The mesh of the stainless-steel expanded metal was  $16 \times 5.5$  mm. The center-to-center spacing of the sheets was 7.5 (6 sheets) or 12.5 mm (4 sheets).

*Operation procedure.* Having adjusted the flow rate of the coolant water in the condenser 7 and turning on the thermostat 8, the preselected power input into the boiler was set. When the flow rate of the condensate measured on the rotameter 9 steadied down the preheater 10 was switched on and set so as to keep the temperature of the reflux at the column inlet about 3 to  $4^\circ\text{C}$  below that of the vapours in the column head. After about two hours when a steady state was reached the samples of the distillate and the bottom product were taken. The analytical methods used were either the densimetry, or the chromatography at higher methanol concentrations. Simultaneously with the sampling the temperatures of the entry and the exit vapour and that of the feed were recorded. The described procedure was repeated at various methanol concentrations in the mixture, various heating intensities and various geometrical arrangements (spacing) of the packing.

*Data processing.* The equilibrium data for the methanol-water mixture were taken over from Othmer<sup>7</sup> and have been used by Kašánek<sup>8</sup> and Hartman<sup>9</sup>. The results of the latter two authors are used for comparison with the present investigation.

The height of a transfer unit was calculated from the familiar formula

$$\text{HTU} = \frac{H}{\int_{y_1}^{y_2} \frac{dy}{y^+ - y}} \quad (1)$$

The integral in Eq. (1) was evaluated numerically on a computer using a Simpson routine. The interval between  $y_1$  and  $y_2$  was divided evenly into 100 steps and the equilibrium concentration  $y^+$  was calculated from the relation due to Prahl<sup>10</sup>

$$y^+ = \frac{Cx(A - x)}{Cx(A - x) + (1 - x)(B + x)} \quad (2)$$

To improve the fit of Eq. (2) the concentration domain was divided into two parts: one between  $x = 0$  and 0.55 and the other between 0.55 and 1. Thus the maximum deviation was kept below 0.15 mol%, while the maximum deviation of the van Laar relation amounts to 0.45 mol%.

## RESULTS

From plots in Figs 2 and 3 it is apparent that the HTU at a given intensity of heating in the boiler decreases up to a concentration of about 30–40 mol% of methanol in the mixture.

It is known from the literature that the efficiency of almost all types of distillation equipment depends on mixture composition. This relation may have extremes or, in other cases, it is a monotonous function. This fact is caused by several effects the most important of which is apparently the difference in surface tension of pure components. From this standpoint the systems may be

divided into three groups<sup>11</sup>: The positive systems with the low-boiling component having lower surface tension. The negative systems with opposite properties and neutral ones where neither of the components exhibits substantially different surface tension or if the relative volatility and hence the surface tension gradients are very small.

The system dealt with here is a positive one with very conspicuous difference of the surface tension. According to Boyes and Ponter<sup>12</sup> the contact angle on copper decreases with increasing methanol concentration from 72.9° for pure water to 2° for

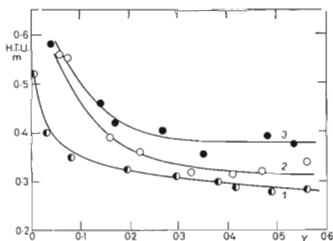


FIG. 3

A Plot of HTU against Mole Fraction of Methanol in Vapour Entering the Column

Expanded metal  $16 \times 5.5$  mm mesh, 7.5 mm spacing 1 3355–4125  $\text{kg/m}^2$  h, 2 7195 to 8310  $\text{kg/m}^2$  h, 3 10290–12600  $\text{kg/m}^2$  h.

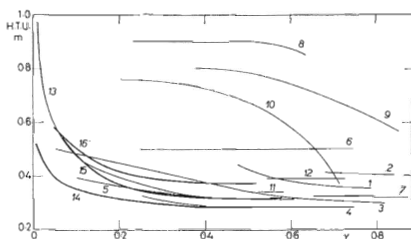


FIG. 4

A Comparison of the Efficiency of the Packing Made of Expanded Metal with Selected Plates<sup>8,9</sup>

1 Uniflux 2000–6500  $\text{kg/m}^2$  h; 2 turbogrid 4200–500  $\text{kg/m}^2$  h; 3 sieve plate I 1200–4500  $\text{kg/m}^2$  h; 4 sieve plate II 1700–3800  $\text{kg/m}^2$  h; 5 ripple 4000–6000  $\text{kg/m}^2$  h; 6 ripple 3500 to 3800  $\text{kg/m}^2$  h; 7 bubble-cap 3400–4600  $\text{kg/m}^2$  h; 8 similar type Kittel 6600–7400  $\text{kg/m}^2$  h; 9 similar type Kittel 5500–6000  $\text{kg/m}^2$  h; 10 similar type Kittel 4300–4500  $\text{kg/m}^2$  h; 11 similar type APV West 2500–2700  $\text{kg/m}^2$  h; 12 similar type APV West 4220–4700  $\text{kg/m}^2$  h; 13 slot plate (Hartman) 2075–2725  $\text{kg/m}^2$  h; 14 expanded metal  $16 \times 5.5$  mm mesh, 7.5 mm spacing, 3460–4560  $\text{kg/m}^2$  h; 15 expanded metal  $16 \times 5.5$  mm mesh, 7.5 mm spacing, 7420–8520  $\text{kg/m}^2$  h; 16 expanded metal  $16 \times 5.5$  mm mesh, 7.5 mm spacing, 9600–12390  $\text{kg/m}^2$  h.

30.5 mol% methanol solution. Above 60 mol% methanol concentration the contact angle is zero. The dependence of the contact angle on the methanol concentration in the mixture is very similar to that of the HTU. One may thus assume that the lowered efficiency is directly associated with the impaired wetting of the packing which was confirmed also by visual observation. Comparing the wetting of the packing on the top and the bottom of the column under low methanol concentrations in the vapour entering the column one can clearly see the favourable wetting conditions on the top of the column in contrast to the tendency to film breakdown and drop formation near the bottom.

Further it can be seen from Figs 2 and 3 that the HTU increases with increasing vapour velocity and it is lower for closer spacing of the sheets for all three power inputs of the heater. The decrease of the HTU with decreasing spacing, *i.e.* with increasing number of the sheets and the area of the packing, can be explained by increased hold-up and hence the residence time of liquid in the column.

Fig. 4 is a comparison of the HTU achieved on our packing with those achieved on some other types of distillation columns<sup>8,9</sup>. At a first sight it is apparent that the HTU achieved on the flat packing at higher vapour and liquid velocities compares favourably with the lowest values achieved on other types of columns. Moreover, the optimum operating range for some types of plates covers a rather narrow range of flow rates.

Comparing the results achieved in this work with those on flat packings mentioned in the introduction it is clear that the new structure of the sheets and the way of wetting has brought about a dramatic improvements of the packing efficiency.

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